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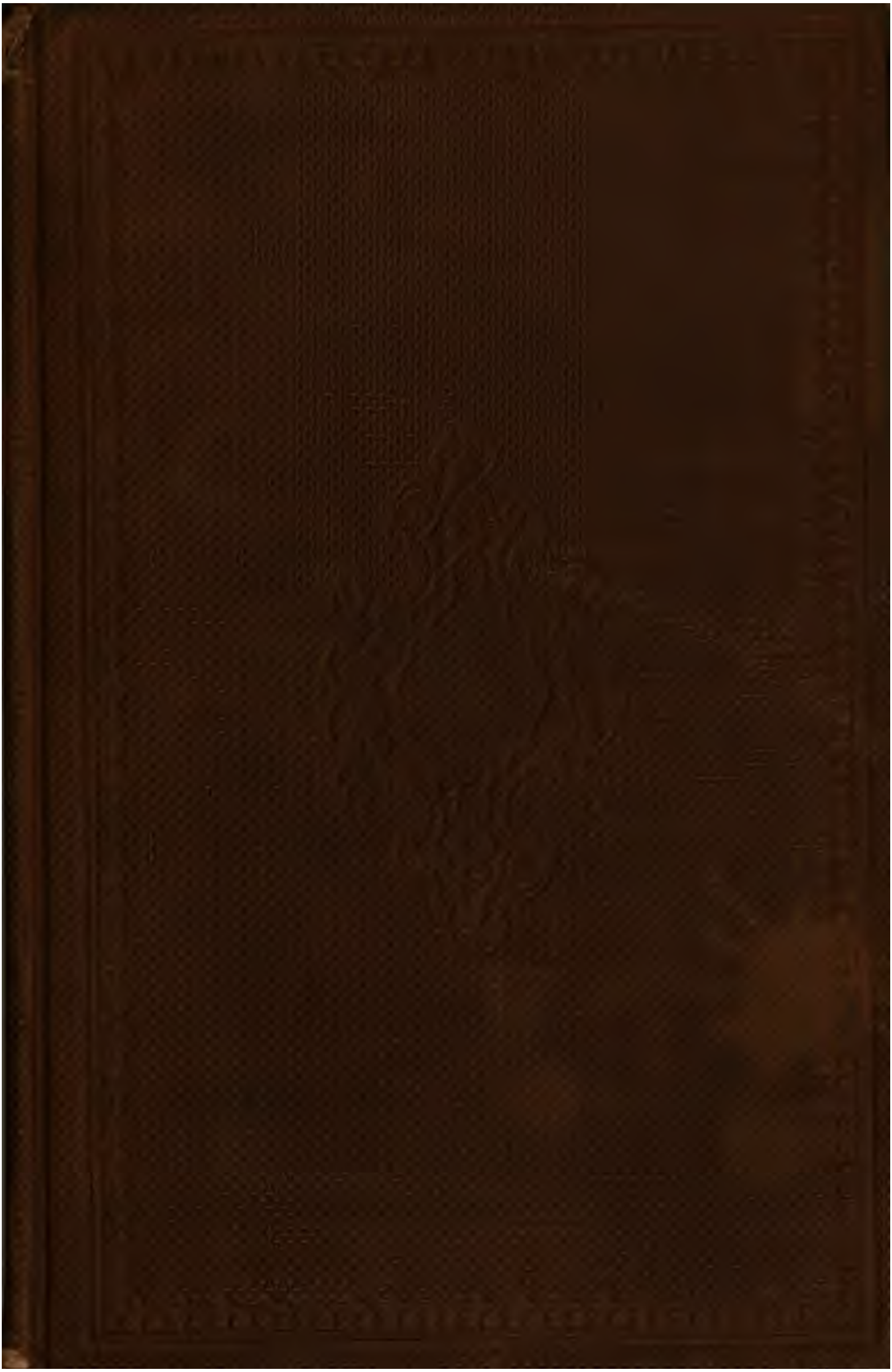

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SCIE ARY

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Cambridge.

L. S. S.

AN INTRODUCTION

TO

PRACTICAL CHEMISTRY.

AN INTRODUCTION

TO

PRACTICAL CHEMISTRY,

INCLUDING ANALYSIS.

BY

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WITH NUMEROUS ILLUSTRATIONS.

THIRD AMERICAN,
FROM THE FOURTH AND REVISED ENGLISH EDITION.

PHILADELPHIA:
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1864.

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Chas. E. Munroe,
of Cambridge.

"Facts are the materials of Science; but all facts involve Ideas. Since, in observing Facts, we cannot exclude Ideas, we must, for the purposes of Science, take care that the Ideas are clear, and rigorously applied."

Whewell, *Philosophy of the Inductive Sciences*, vol. i. p. xxxvii.

PHILADELPHIA:
COLLINS, PRINTER.

PREFACE

TO THE FOURTH EDITION.

IN this Edition, some important alterations have been made in that portion of the work which is devoted to Chemical Analysis. A division of the substances to be detected, into metallic and non-metallic bodies, has been adopted in the outset, instead of the older division into bases and acids, by which, it will be found, much ambiguity is avoided.

In the systematic course of Qualitative Analysis, a wider range has been taken, so as to admit cases not provided for in preceding editions, and more care has been employed to insure the detection of the constituents of such complex compounds as are met with in the analysis of ores and minerals. This part of the work has been entirely re-written.

The chapter referring to the use of the blowpipe has been enlarged, so as to include the detection of all the ordinary elements which admit of recognition by the blowpipe, and a systematic course for the examination of unknown substances has been added.

The method described in the section on Alkalimetry has been modernized, and the determination of iron by means of permanganate of potash has been added, in order to afford the student, in this chapter, a fair illustration of the methods of Volumetric Analysis, which are now so generally employed by the chemist.

Some alterations have been made in the examples of quantitative analysis; and a concluding chapter has been added, containing directions for improvising reagents, or substitutes for them, when they cannot be otherwise procured.

King's College, London,
July, 1861.

PREFACE

TO THE FIRST EDITION.

AMONG the many recent and valuable works on Chemistry, I am not aware of one having for its special object to explain, and render simple to the beginner, the various processes employed in analysis, or which have been devised for the illustration of the principles of the Science. Most of them contain much that is superfluous for the general student, who has but a limited time to devote to the subject; while they are wanting in those explanatory details, without which he must often fail to understand the rationale of the operations through which he is conducted.

It is with a wish to supply this deficiency, and at the same time to furnish a text-book for my own classes, that the present little work has been written; and as it is intended for the use of those who have made but little progress in the Science, my endeavor has been, throughout, to make everything as simple and intelligible as possible. The employment of complicated or expensive apparatus has been almost wholly avoided.

The outline of most of the First Part was arranged some years ago by my friend Professor Miller (at that time Demonstrator of Chemistry in King's College), for the use and direction of the class of Chemical Manipulation, then first established to supply a growing demand, and to meet the requirements of the University of London, and some of the other examining Boards of the Metropolis. In the compilation of the Second and Third Parts, I have been much indebted to the excellent works of Rose, Fresenius, Parnell, and others; I must also here thank my colleague, whose name I have already mentioned, for many valuable suggestions, and for his kindness in revising the proof-sheets, without which assistance many errors would have crept in, and rendered the book less worthy of the student's confidence.

JOHN E. BOWMAN.

KING'S COLLEGE, LONDON,
September, 1848.

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PRACTICAL CHEMISTRY.

PART I.

CHAPTER I.

INTRODUCTORY—SYMBOLS—EQUATIONS—GENERAL RULES.

1. So essentially is chemistry an experimental science, and so almost exclusively is it built up of facts which have been elucidated by experiment, that without experimental illustrations it would be quite impossible to teach or to study it with any great amount of success. It is not enough, however, for the student to see experiments performed by others; he must, if he would master even the general principles of chemistry, learn to make experiments himself; and he will probably be surprised how much more easily he will retain in his recollection those phenomena (as well as the principles they illustrate) which his own hands have been the means of producing. This is especially the case when he is enabled, while operating in the laboratory, to learn and study the theory of the changes which take place under his direction.

2. With the view of enabling the beginner to do this as much as possible, I have in the following pages explained, by means of chemical symbols and equations, nearly the whole of the changes and decompositions which take place in the experiments described. The symbols which I have made use of are those now almost universally adopted by chemists; and it will be seen by the following table, that they consist, for the most part, of the first letter or two letters of the Latin names of the elements which they represent.

Table of the most important Elementary Substances (arranged alphabetically), showing their symbols, atomic weights,[1] *and the composition of some of their compounds.*

Name.	Symbol.	Atomic weight.	Compounds.
Aluminum	Al	13.7	Al_2O_3 Alumina.
Antimony. (Stibium.)	Sb	122	SbO_5 Antimonic Acid.
Arsenic	As	75	AsO_3 Arsenious acid.
Barium	Ba	68.5	BaO Baryta.
Bismuth	Bi	210	$BiCl_3$ Chloride of Bismuth.
Boron	B	10.9	BO_3 Boracic acid.
Bromine	Br	80	BrO_5 Bromic acid.
Calcium	Ca	20	CaO Lime.
Carbon	C	6	CO_2 Carbonic acid.
Chlorine	Cl	35.5	HCl Hydrochloric acid.
Chromium	Cr	26.3	CrO_3 Chromic acid.
Cobalt	Co	29.5	CoO Oxide of cobalt.
Copper. (Cuprum.)	Cu	31.8	CuO Black oxide of copper.
Fluorine	F	19	HF Hydrofluoric acid.
Gold (Aurum.)	Au	196.7	$AuCl_3$ Terchloride of gold.
Hydrogen	H	1	HO Water.
Iodine	I	127	HI Hydriodic acid.
Iron	Fe	28	Fe_2O_3 Sesquioxide of iron.
Lead (Plumbum.)	Pb	103.5	PbO Oxide of lead.
Magnesium	Mg	12.2	MgO Magnesia.
Manganese	Mn	27.5	MnO_2 Black oxide of manganese.
Mercury (Hydrargyrum.)	Hg	100	Hg_2Cl Calomel. $HgCl$ Corrosive sublimate.
Nickel	Ni	29.5	NiO Oxide of nickel.
Nitrogen	N	14	NH_3 Ammonia.
Oxygen	O	8	HO Water.
Phosphorus	P	31	PO_5 Phosphoric acid.
Platinum	Pt	98.6	$PtCl_2$ Bichloride of platinum.
Potassium	K	39	KO Potash.
Silicon	Si	14	SiO_2 Silicic acid.
Silver (Argentum.)	Ag	108	$AgCl$ Chloride of silver.
Sodium (Natrium.)	Na	23	NaO Soda.
Strontium	Sr	43.8	SrO Strontia.
Sulphur	S	16	HS Hydrosulphuric acid.
Tin (Stannum.)	Sn	59	SnO_2 Binoxide of tin.
Zinc	Zn	32.5	ZnO Oxide of zinc.

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[1] The second decimal figure has been omitted, in order that the numbers may be more readily committed to memory.

3. Each of these symbols expresses one equivalent or atom of the substance which it represents. Thus, H stands for one atom or equivalent of hydrogen; Cu for an equivalent of copper; *Hg* for one of mercury.[1]

When a small figure is placed to the right of a symbol, rather below the line, it means that there is that number of equivalents of the substance present. Thus, Pb_2 means two equivalents of lead; O_5 five equivalents of oxygen; H_{10} ten equivalents of hydrogen.

Two or more symbols placed together signify that the elements which they represent are chemically united, without expressing any view of the *constitution* of the compound—that is, of the manner in which the elements are grouped. Thus *HO* stands for water, which is a compound of one equivalent of hydrogen and one of oxygen; SO_3 represents anhydrous sulphuric acid, composed of one equivalent of sulphur and three of oxygen; $C_{12}H_{10}O_{10}$ represents starch, which consists of 12 equivalents of carbon, 10 of hydrogen, and 10 of oxygen, chemically combined together.

When symbols are separated by a comma, they are intended to represent the manner in which the elements are grouped together in the compound. Thus, sulphate of potash, which contains one equivalent of potassium, one equivalent of sulphur, and four of oxygen, is written KO,SO_3, implying that it may be regarded as a compound of potash (KO) with anhydrous sulphuric acid (SO_3), this view being the most convenient for the explanation of most of the chemical changes in which this salt takes part.

When the sign + is interposed, it indicates that the substances between which it is placed are less intimately united. Thus, in crystallized carbonate of soda (NaO,CO_2+10Aq), we have sodium and oxygen in the soda, and carbon and oxygen in the carbonic acid, combined in the closest and strongest manner; the soda and carbonic acid thus formed are separated by a comma, showing that they are held together by what we may here call the second degree of affinity; while the 10 equivalents of water of crystallization, separated by the sign +, are held by a much weaker force, so feeble indeed that a very moderate heat is sufficient to expel them.

---

[1] **The student is reminded that atom and equivalent are not necessarily synonymous.**

The sign + is used also to separate the symbols of substances which are entirely disunited, thus the addition of hydrochloric acid to carbonate of lime is represented by CaO,CO_2+HCl.

A large figure placed immediately before a symbol, multiplies all the symbols as far as the next comma or + sign. Thus, in the common phosphate of soda ($2NaO, HO,PO_5$) there are two equivalents of soda, one of water and one of phosphoric acid combined together. If a large figure were placed before the whole formula inclosed in brackets, thus, $5(2NaO,HO,PO_5)$, it would represent 5 equivalents of the entire salt.

4. It is really wonderful how much these little symbols are capable of expressing, and how often and completely they assist in simplifying and rendering intelligible even the most complicated chemical changes; for besides the information they convey relative to the composition of the substances which they express, they can be so combined in the form of equations, as to show in the most perfect manner, the various compounds which result during chemical decompositions. For this purpose, the symbols of the substances employed are placed together so as to form one side of the equation; on the other side are placed those of the substances which are produced during the decomposition; and as no atom of matter is lost during these transformations, it necessarily follows that the value of both sides of the equation must be equal. For example, the decomposition of carbonate of lime by hydrochloric acid may be thus represented:—

$$CaO,CO_2+HCl=CaCl+HO+CO_2.$$

Here we place the symbols of carbonate of lime and hydrochloric acid on one side, and on the other those of chloride of calcium, water, and carbonic acid, which are produced during the decomposition; and it will be observed that on each side there are exactly the same number of equivalents, viz., 1 of calcium, 3 of oxygen, 1 of carbon, 1 of hydrogen, and 1 of chlorine.

5. I have ventured to introduce a slight modification of the usual mode of printing the symbols, which will enable the student to see at a glance whether the substances expressed are in the solid, liquid, or gaseous form.

Those in the solid state are printed in strong Roman type, as $\mathbf{Pb}$, lead. Liquids or substances in solution, are printed in strong Italics, as $\boldsymbol{HO}$, water; and gases or vapors are represented by fine hair letters, as H, hydrogen, HO, steam.

Thus in the above equation, liquid hydrochloric acid ($\boldsymbol{HCl}$) is poured on solid carbonate of lime ($\mathbf{CaO,CO_2}$); chloride of calcium ($\boldsymbol{CaCl}$) is formed, which remains in solution, together with carbonic acid ($\mathrm{CO_2}$) which passes off in the gaseous form.

6. It is very important that the student should at once begin to make careful notes of all the experiments he engages in. He should endeavor to do this in as concise and methodical a manner as possible, and he will find it very advantageous to make use of symbols in describing the substances he employs, and the changes which they undergo: he will thus be able to record much in a small space, and at the same time he will be making himself familiar with the composition of the substances with which he is experimenting.

7. When, as is often the case, especially in analytical experiments, there are several solutions and precipitates either filtering, digesting, or waiting till the operator has leisure to attend to them, it is necessary to mark them in some way, to prevent confusion. This is easily done with small pieces of gummed paper, on which a letter or number may be written, corresponding with a similar reference mark in the note book.

8. The student will soon learn by experience that he cannot be too methodical in his operations, or too careful in cultivating habits of neatness and cleanliness. The presence of a little saline or other impurity in a glass, owing to careless washing, or a little extraneous matter having been allowed to find its way into a bottle or test-

tube, may retard or spoil the result of whole days of labor.

"Much as the chemist may soil his fingers during his experimental occupations, he will soon learn the great importance of cleanliness to the success of his experiments. The regular course of his operations causes many kinds of matter to pass in succession through his hands; and many of the substances, which by mixture have exhibited the phenomena they were competent to occasion, and so far answered the purpose of the experiment, then become mere useless dirt. Their dismissal and entire removal when thus circumstanced become necessary, that they may not contaminate other bodies; and are as imperatively required, as was the care previously bestowed to prevent their contamination from extraneous matter.

"It is this rapid change in the character and relation of the substances with which the chemist works, that makes a constant attention to cleanliness essentially necessary. The very bodies which at one moment are carefully retained in vessels that have previously been cleansed with the most scrupulous attention, become the next in the situation of so much dirt, from which the vessels must be cleansed as perfectly and carefully, before they can be fit for another experiment, as they were for the reception of the now rejected matter. The results of numerous experiments relative to testing bodies in solution by reagents, are, in many cases, dependent on the employing of clean vessels. For instance, a portion of water examined in glasses which have been carelessly washed may occasion a slight precipitate with nitrate of silver or chloride of barium, and thus seem to contain a chloride or a sulphate when the cause of the precipitate may be nothing more than portions of salts adhering to the vessel.

"In the same manner the purity of an acid or a test is not unfrequently affected by the state of the bottle containing it, or by the dirty condition of glass rods dipped into it, or of the funnels through which it has been poured or filtered, or of the vessels used in its transference; and sometimes it is contaminated by laying the stopper of the bottle containing it in a dirty place. Nor is it only that kind of dirt or impurity which gives an evident tinge to what it adheres to, that is to be avoided, but also numerous colorless substances, as salts, solutions, &c.; and in a word, anything which differs from the principal substance itself, and is at the same time liable to be dissolved or mixed with it.

"In consequence of these liabilities, and their interference with experiments, it should be established as a general rule in the laboratory, that no apparatus, nor any vessel (except such as may be destined to a particular use, and is as convenient when with a little previously adhering matter as if it were clean), be put away in a dirty state. All vessels or instruments when resorted to should be found fit for the nicest experiment to which they are applicable. Glass rods or stirrers should be preserved in a clean place; glasses, on a clean shelf; and stoppers, when taken out of bottles, should be laid upon clean surfaces. These attentions and regulations will be found always useful, at times essential; and they are generally more requisite and influential in minute chemistry, than in large experiments."[1]

9. It is easy to clean even the dirtiest vessel, provided it has not been allowed to remain long with the impurities adhering to it; this, indeed, should never be permitted, and is readily avoided by making it a rule never to leave work for the day until the whole of the soiled apparatus has been thoroughly washed, and left to drain during the night, ready for wiping the next morning. For most purposes of cleaning, water will be found sufficient, especially when the dirt is still moist; and when mere rinsing does not remove it, gentle friction with moist tow and coal ashes or sand will, in most cases, prove effectual. When the form of the vessel to be cleaned is such as will not allow the introduction of the hand (as flasks, test-tubes, &c.), a small bottle-brush, or a piece of stick or wire, having a little tow wrapped round the end, will be found very convenient. Glasses or basins that have been set aside to drain should, before use, be wiped with a

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[1] Faraday's Chemical Manipulation, p. 523.

dry clean cloth, to remove any adhering particles of dust or moisture. Bottles or flasks, when required to be perfectly dry inside, may, after most of the water has been removed, be easily dried by warming them gently, and drawing air into them through a glass tube, with the mouth; in this way the water is converted into vapor, which is quickly removed by the current of comparatively dry air.

When a glass or dish is greasy, it should be first wiped as clean as possible with tow or a dry cloth, then moistened with a little strong potash, and, lastly, well washed and rinsed with water. When the dirt to be removed is resinous, also, or tarry, the application of strong potash or sulphuric acid will generally act upon it in such a way, that subsequent washing with water, together with gentle friction with sand, will render it quite clean. It often happens, especially when a glass has been allowed to dry in a dirty state, that an insoluble crust is formed on the surface which is very difficult of removal by mechanical means, but readily yields on the application of a few drops of hydrochloric or some other acid. An instance of this is afforded by solutions of lime, which, on exposure to the air, frequently deposit a crystalline sediment of carbonate of lime, which adheres strongly to the glass, but instantly dissolves on the addition of the acid.

10. When thrown upon his own resources, the student will often find it of the utmost value to be able to substitute, in default of more perfect apparatus, the common things used in domestic life, which are to be found in every house, such as glasses, plates, cups, saucepans, &c. When in addition to these he has at his command a blowpipe, a small piece of platinum foil and wire, a flask or two, a funnel, and a little glass tubing of different sizes, he will, with the exercise of a little ingenuity and contrivance, be able to go through a very considerable course of experimental chemistry. He may rest assured that it is no disadvantage, but rather the contrary, to be thus compelled to devise and construct for himself rude and extemporaneous forms of apparatus; and if he should require encouragement to persevere in spite of the scantiness of his resources, he need only be reminded that the majority

of those whose names shine brightest in the annals of science, have laid the groundwork of their future eminence while placed under the most unfavorable circumstances. So it was with the great Davy;[1] so with Dalton, with Scheele, Faraday, Dumas, Liebig, and many others almost equally illustrious.

"Habits of correct and delicate manipulation very much facilitate experimental inquiries at all times. It is not in difficult researches only that it is desirable, but even in such common operations as testing for lime, or iron, or sulphuric acid, its advantages become manifest; for either time is shortened, or the apparatus considered as necessary is diminished, or effectual substitution is made for those that may be wanting, and thus the experiment becomes easy, where otherwise it would be considered impossible. Besides facilitating such inquiries, it also diminishes the expense both in materials and apparatus, and it produces beneficial habits in the mind, by exercising it both in invention and perception even in this subordinate part of its operation. 'Nothing,' as Dr. Johnson observes, 'is to be considered as a trifle, by which the mind is inured to caution, foresight, and circumspection. The same skill, and often the same degree of skill, is exerted in great and little things.'"[2]

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[1] "His means, of course, were very limited; not more extensive than those with which Priestley and Scheele began their labors in the same fruitful field. His apparatus, I believe, consisted chiefly of phials, wine-glasses, and teacups, tobacco-pipes, and earthen crucibles; and his materials were chiefly the mineral acids and the alkalies, and some other articles which are in common use in medicine."—*Life of Sir H. Davy*, by John Davy, M.D., vol. i. p. 43.

[2] Faraday, op. cit. p. vi.

CHAPTER II.

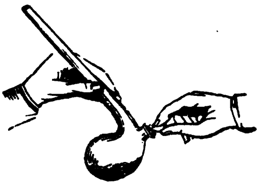
PNEUMATIC CHEMISTRY.

SECTION I.

Preparation of Hydrogen (H).[1]

11. Weigh 300 grains of granulated zinc, and introduce the fragments carefully through the tubulure of the retort, sliding them, not dropping them in, to avoid the risk of breaking the bottom of the retort, which is usually of thin glass, and consequently seldom strong enough to bear a blow without injury.

Fig. 1.



Pour upon the metal four fluid ounces of dilute sulphuric acid, consisting of one part by measure of oil of vitriol (HO,SO_3), and five parts of water.

Effervescence immediately commences, owing to the evolution of the gas, the zinc being rapidly dissolved in the form of sulphate. The gas may be collected in jars filled with water, and inverted upon the shelf of the pneumatic trough (Fig. 2).[2]

Fill five or six gas-cylinders with hydrogen before commencing the following experiments, and set the retort aside upon a list ring.

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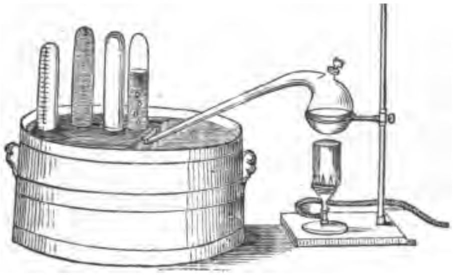
[1] The specific gravity of hydrogen is lower than that of any other form of matter, being only 0.069, that of common air being considered 1.000. 100 cubic inches weigh, at the ordinary temperature and pressure of the air, 2.14 grains, while the same quantity of common air weighs 31.00 grains. The atomic weight of hydrogen is 1, and its combining volume 2.

[2] The preparation of hydrogen, with acid of the prescribed strength, does not require the application of heat.

12. Lift the jar which was first filled off the shelf, still keeping its mouth downwards, and introduce a lighted taper; a slight explosion will probably ensue, because the hydrogen is mixed with atmospheric air previously contained in the retort: showing that *a light must never be applied to a hydrogen apparatus until all the air has been expelled.*

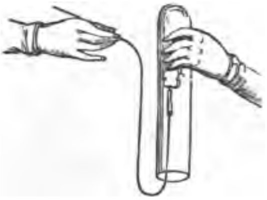
Fig. 2.



Pneumatic Trough.

Repeat the experiment with a jar of hydrogen free from air; the taper is extinguished, for the gas does not support combustion, but the hydrogen takes fire at the mouth of the jar with a slight report, and if the taper be carefully withdrawn, it may be rekindled at the flame of the hydrogen, and this extinction and rekindling of the taper may be repeated until the gas has burnt out.

Fig. 3.

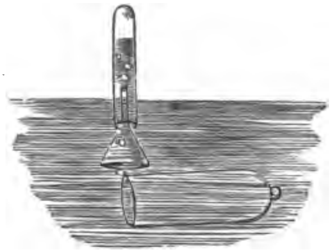


13. Fill a small jar with the gas, and having removed it from the shelf, let it stand for a few seconds with its open end *upwards.* If a lighted taper be now applied (Fig. 6), no combustion will ensue, as the hydrogen will have escaped upwards, on account of its very low specific gravity.

14. Repeat the last experiment, holding the jar with the open end *downwards.* On applying a lighted taper, a

slight explosion will take place, showing that the hydrogen had not entirely escaped as before.

Fig. 4.

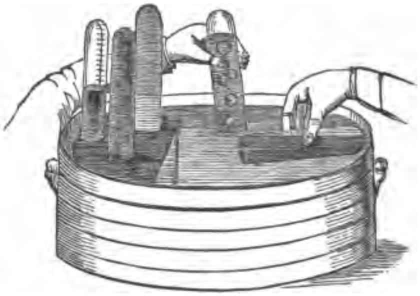


15. Transfer some of the gas from a large jar to a small one, and from this again to tubes, until it can be done without allowing any bubbles to escape. When the gas is to be decanted into a jar or tube which is much narrower, it may be first transferred into a lipped glass; or an inverted funnel may be used, as shown in Fig. 4.

16. Transfer a little of the hydrogen in this way into a graduated jar; measure out two cubic inches, and transfer them into a larger jar.

The graduated jar being filled with water, close its mouth, under the water, with a glass plate, then raise it, with the glass plate beneath, out of the water, and slip the plate gradually aside, so that bubbles of air may enter; measure out, in this way, two cubic inches of air, slide the plate back again so as to close the jar, and decant the air, under water, into the jar containing the two cubic inches of hydrogen; then transfer the mixture into

Fig. 5.



Decanting Gas.

a small strong jar, close it with a glass plate, place it upright upon the table, remove the plate, and apply a lighted taper.

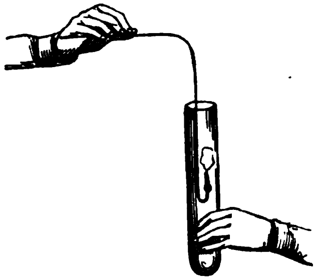
Repeat the experiment a second time, with two cubic inches of hydrogen and four cubic inches of air; and a third time, with six cubic inches of air, and ascertain in which case the loudest detonation takes place.

SECTION II.

Preparation of Carbonic Acid (CO_2).[1]

17. Put 300 grains of marble (CaO,CO_2) broken into fragments about the size of a pea, into a retort, observing the same precautions as were recommended in the preparation of hydrogen (11). Measure out an ounce and a half of hydrochloric acid (*HCl*), dilute it with an equal quantity of water, and pour the mixture upon the marble. The gas is immediately given off, causing brisk effervescence, and it may be collected in jars placed on the shelf of the pneumatic trough, the first jar-full being rejected as impure.

Fig. 6.



$$CaO,CO_2 + HCl = CaCl + HO + CO_2.$$

18. Introduce a lighted taper into a small jar of the gas held with its open end upwards. It is instantly extinguished; and as the carbonic acid remains some time in the jar, on account of its high specific gravity, the taper may be extinguished repeatedly in the same jar-full of gas.

19. Pour a little lime-water (CaO,HO) into a test glass,

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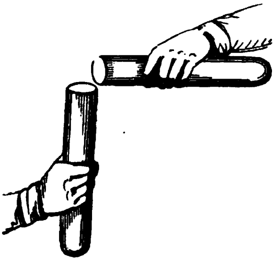
[1] The specific gravity of carbonic acid is 1.529 (air being 1.0), 100 cubic inches weighing 47.30 grains. Its atomic weight is 22; and its combining volume 2. At a temperature of 60° water dissolves about its own bulk of carbonic acid.

and thence into a jar filled with the gas,[1] closing the mouth of the jar with a glass plate, and agitating the gas and liquid together. The lime-water almost immediately becomes milky, owing to the formation of carbonate of lime (CaO,CO) which is insoluble in water. Pour a little of this milky fluid into another jar of carbonic acid; on closing with a glass plate and shaking violently, the milkiness will disappear, for the excess of carbonic acid converts the insoluble carbonate into the soluble bicarbonate of lime.

If a few drops of hydrochloric acid (HCl) be added to the other portion, the carbonate of lime is decomposed, and the milkiness disappears, chloride of calcium being formed, which is soluble in water.

$$(CaO,CO_2+HCl=CaCl+HO+CO_2).$$

Fig. 7.



20. Having filled a jar with the gas, pour it into another jar somewhat smaller (Fig. 7); this is easily effected, owing to the high specific gravity of carbonic acid. Test its presence in both jars with lime-water (19), and by its power of extinguishing a taper (18).

21. Allow a little of the gas to escape, under water, from a jar filled with carbonic acid, and when a little water has thus entered the jar, close it with the palm of the hand, remove it from the trough, and shake violently to and fro; a portion of the carbonic acid will be absorbed by the water, and on plunging the jar again beneath the surface and removing the hand, more water will enter to supply the place of the gas which had been absorbed; on adding some lime-water to the water in the jar, the presence of carbonic acid will be rendered evident.

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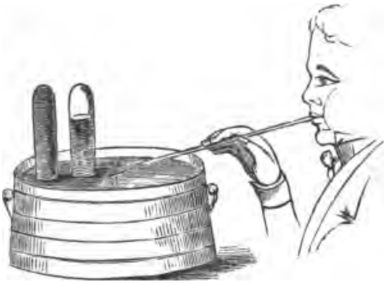
[1] If the lime-water were poured from the bottle at once into the gas-jar, a quantity of the carbonic acid would enter the bottle and render the whole of the lime-water turbid.

22. By means of a narrow tube open at both ends, fill a jar over the pneumatic trough, with air from the lungs.

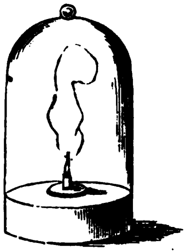
Fig. 8.



Test it with a lighted taper, and observe that it causes an abundant precipitate in lime-water, owing to the presence of carbonic acid (19).

23. Invert a jar filled with common air over a lighted taper floating on the water of the pneumatic trough (Fig. 9); observe that it soon burns dimly, and is shortly extinguished, the water at the same time slowly rising in the jar. The absorption of air is here owing to the disappearance of part of the atmospheric oxygen, which combines with the hydrogen and carbon of the burning wax, forming water which condenses upon the sides of the jar, and carbonic acid gas which is equal in volume to the oxygen consumed to form it, and extinguishes the taper long before the whole of the oxygen is exhausted.

Fig. 9.



When the combustion is over, invert the jar, and test the air contained in it with lime-water for carbonic acid (19).

Expose a few drops of lime-water to the air upon a clean glass plate, and observe the film of carbonate of lime produced after a few minutes by the atmospheric carbonic acid.

SECTION III.

Preparation of the Binoxide of Nitrogen (NO_2).[1]

24. Put 300 grains of copper turnings into a retort, and pour upon it three ounces of dilute nitric acid (HO, NO_5) containing equal measures of the strong acid and water. Decomposition immediately commences on applying a gentle heat, and the binoxide is formed by the action of the copper on a portion of the nitric acid, thus:—

$$3Cu+4(HO,NO_5)=3(CuO,NO_5)+NO_2+4HO.$$

The gas which is first formed becomes orange, owing to its conversion into peroxide of nitrogen (NO_4) by combining with the atmospheric oxygen contained in the retort ($NO_2+2O=NO_4$).

Before removing the flame from under the retort, be careful to take the neck out of the water, or the latter will be forced up into the retort as it cools, and may either break or overbalance it.

Transfer a little to a jar and test it with a taper;[2] observe the orange fumes of peroxide of nitrogen which are instantly produced whenever the gas mixes with the air.

25. Measure 2½ cubic inches of the gas in a graduated cylinder, and transfer it to another jar over the pneumatic trough; then measure off an equal volume of atmospheric air, and add it, by decantation, to the binoxide. When the orange fumes have disappeared, owing to the absorption of the peroxide of nitrogen by the water, transfer it again to the graduated jar, and observe the volume of the mixture, noticing accurately the difference between this and the sum of the original volumes employed before mixing. This experiment should be repeated three or four times, and if the results in each

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[1] The specific gravity of binoxide of nitrogen is 1.039, 100 cubic inches weighing 32.22 grains. Its atomic weight is 30, and its combining volume 4.

[2] Pure binoxide of nitrogen extinguishes a taper, but the gas obtained by the above process generally contains nitrous oxide (NO) which enables it to support combustion.

case agree pretty closely, take the average of the experiments, and the amount of condensation, divided by three, should give nearly the quantity of oxygen contained in the atmospheric air employed. One equivalent of binoxide of nitrogen occupying four volumes, when combined with two equivalents of oxygen occupying two volumes, forms one equivalent of peroxide of nitrogen (NO_4) which is absorbed by the water; consequently, one-third of the gas absorbed consists of atmospheric oxygen. If the experiment be carefully performed, the absorption will be found to be equal to about three-fifths of the volume of common air employed, the proportion of oxygen contained in it being one-fifth.

Though the results obtained in this way are not very accurate, owing to the formation of other oxides of nitrogen, the experiment is useful as an exercise in the measurement of gases.

SECTION IV.

Preparation of Olefiant Gas (C_4H_4).[1]

26. Pour into a retort six fluid drachms of alcohol (C_4H_5O,HO) and add to it in small portions an ounce and a half of strong sulphuric acid (HO,SO_3), gently agitating the mixture after each addition. Apply heat, and take care that the black froth which is formed towards the close of the operation does not boil over. Collect the gas in jars over the pneumatic trough.

Observe the odor of ether possessed by the bubbles at the beginning of the process, and that of sulphurous acid towards the end. The olefiant gas has a peculiar odor of garlic.

27. Examine a small jar-full with a taper and observe that, though the taper is extinguished, the gas burns with a bright white flame, a property which constitutes its great value as the chief illuminating constituent of coal-gas.

28. When mixed with an equal volume of chlorine (Cl)

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[1] The specific gravity of olefiant gas is 0.978. Its atomic weight is 28, and its combining volume 4.

the two gases combine, forming a heavy oily compound, called Dutch liquid ($C_4H_4Cl_2$).

$$(C_4H_4)+2Cl=C_4H_4Cl_2.$$

The oil collects in drops on the sides of the jar and on the surface of the water, while the gases are gradually absorbed.

Olefiant gas derives its name from the circumstance of its forming the oily compound.

29. Mix together one volume of olefiant gas and two volumes of chlorine; close the jar with a glass valve, and quickly remove it from the pneumatic trough. Apply a light to the mixed gases, and observe the dense cloud of carbonaceous matter that is formed as the combustion gradually passes down the jar, hydrochloric acid being at the same time produced.

$$C_4H_4+4Cl=4HCl+4C.$$

SECTION V.

Preparation of Carbonic Oxide (CO).[1]

30. Carbonic oxide is prepared by the action of strong sulphuric acid (HO,SO_3) on oxalic acid (HO,C_2O_3+2Aq). When a mixture of the two acids is warmed, the oxalic acid is resolved into carbonic acid, carbonic oxide, and water, which latter unites with the sulphuric acid.

$$HO,C_2O_3+2Aq=CO_2+CO+3HO.$$

The carbonic oxide is purified from the carbonic acid by passing it through a solution of potash or milk of lime.

$$CO_2+CO+KO=KO,CO_2+CO.$$

31. Adapt a cork to a wide-mouthed bottle capable of holding half a pint of water, and fit to it two tubes, one of which (*a*, Fig. 10), should be about half an inch in diameter, straight, and sufficiently long to reach nearly to the bottom; the other, *b*, should only just pierce through the cork, and should be bent so as to deliver the gas, as

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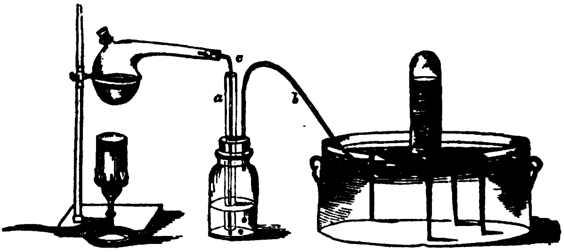
[1] The specific gravity of carbonic oxide is .967. Its atomic weight is 14.0 and its combining volume 2.

shown in the figure; the diameter of this tube need not be more than about $\frac{1}{3}$ of an inch.

Fig. 10.



Preparation of Carbonic Oxide.

The beak of the retort may now be fitted with a cork, which should be bored to allow the bent tube *c* to pass through it; and care must be taken that this tube is sufficiently small to slide easily down the tube *a*, and long enough to reach to the bottom of the bottle.

Four ounces of a tolerably strong solution of potash (KO) may now be introduced into the bottle.

32. Charge the retort with 180 grains of crystallized oxalic acid (HO,C_2O_3+2Aq) and two fluidounces of strong sulphuric acid (HO,SO_3). On applying a gentle heat, the gas is given off, the first portions of which must be rejected as impure, and then two or three jars-full may be collected over the pneumatic trough before the bottle containing the potash is connected with the retort. The gas thus obtained is a mixture of carbonic acid and carbonic oxide (30).

33. Having collected two or three jars-full of the mixed gases for comparison, adapt the bent tube *c* to the mouth of the retort, and proceed to purify the gas from carbonic acid, by passing it through the alkaline solution in the bottle. Pure carbonic oxide may then be collected.

34. Agitate a little lime-water with a jar full of the unpurified gas; the presence of carbonic acid is shown by the formation of carbonate of lime (19).

35. Repeat the experiment with a jar-full of the purified gas. No precipitate ought now to appear.

36. Apply a lighted taper to a jar-full of the impure gas, and observe the characteristic pale blue flame with which the carbonic oxide burns.

37. Do the same with a jar of the pure gas; the flame is brighter than when carbonic acid was present.

38. Pour a little lime-water into the jar used in the last experiment immediately after the combustion of the gas. The white precipitate which now appears, and which was not formed when the same gas was tested previous to the combustion, shows the result of that process to have been the formation of carbonic acid.

$$CO + O = CO_2.$$

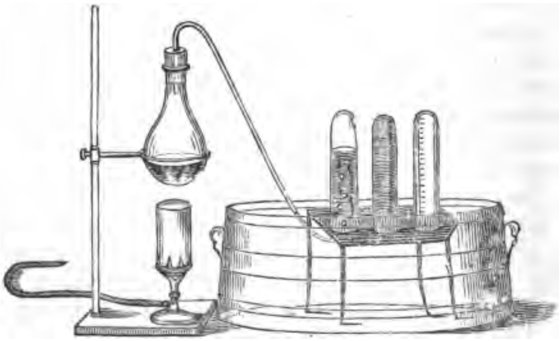
SECTION VI.

Preparation of Oxygen (O).[1]

39. Adapt a bent tube of the form shown in the figure, to a small hard glass flask,[2] by means of a perforated cork.

Then weigh 100 grains of dried chlorate of potash (KO,ClO_5), mix it with 20 grains of black oxide of manganese (MnO_2), and place the mixture in the flask;

Fig. 11.



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[1] The specific gravity of oxygen is 1.1057, 100 cubic inches weighing 34.20 grains. Its atomic weight is 8, and its combining volume 1.

[2] For this and similar processes, a Florence oil-flask will be found most convenient.

adjust the tube so as to deliver the gas under the shelf of the pneumatic trough (Fig. 11), and apply the heat of a lamp.

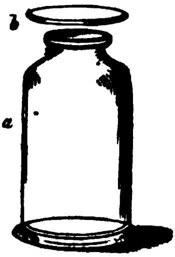
40. The chlorate of potash is thus decomposed, and gradually gives off the whole of its oxygen, which passes out through the tube, while chloride of potassium (KCl) remains in the flask, together with the oxide of manganese, which is not decomposed during the process.[1]

$$KO,ClO_5 = KCl + 6O.$$

The first portion of the gas should be rejected as impure, being mixed with the common air contained in the flask and tube.

41. The jars used for the following experiments should be open both at the top and bottom, the edges of both being ground smooth, so as to be closed air-tight with a glass valve *b* (Fig. 12).

Fig. 12.



42. Fill a jar with the gas, and introduce a glowing taper; it will instantly burst into flame, and burn with great brilliancy, until most of the oxygen is exhausted by combining with the carbon and hydrogen of the wax.

43. Introduce into another jar of the gas a small piece of ignited charcoal, attached to the end of a wire. It bursts into vivid combustion, combining with the oxygen, and forming carbonic acid (CO_2), the presence of which may be proved by agitating a little lime-water in a jar (19).

44. Repeat the experiment with a small coil of thin iron wire, to which a little charcoal or amadou should be attached and ignited, for the purpose of heating the iron sufficiently to cause it to burn. The iron combines with the oxygen, forming the black oxide (Fe_3O_4), fused globules of which drop to the bottom, and should be received in water, as they are so intensely

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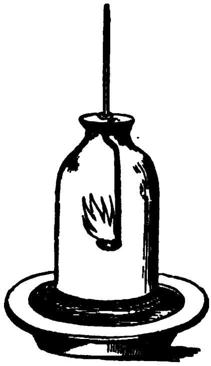
[1] The oxide of manganese is here used, because it is found that, when thus mixed, chlorate of potash gives off its oxygen with much greater facility and at a lower temperature than when heated alone.

hot as to fuse into the glaze of a plate if allowed to fall upon it.

45. Place a fragment of sulphur about the size of a pea in the deflagrating spoon, set it on fire by holding it over a lamp, and introduce it into a jar of the gas; the sulphur burns with a brilliant blue flame, combining with the oxygen, and forming sulphurous acid (SO_2).

Fig. 13.



46. Mix together two volumes of hydrogen and one of oxygen, and with the mixture fill a small jar or tube, which for this experiment should be made of thick glass. On applying a light, the gases combine with a loud explosion, forming water. $H + O = HO$.

SECTION VII.

Preparation of Gases which are soluble in Water.

47. Although in the preparation of many of the common gases it is most convenient to collect them over water, still there are many cases in which this method is inapplicable, as when the gas is to any considerable extent soluble in water. It is usual in such cases, especially when great purity is necessary, to collect them in tubes or jars over mercury, which is not acted upon by the majority of the gases. For common purposes, however, some of them may be collected by the displacement of common air in dry bottles, and the more the gas differs in density from atmospheric air, the more is this method applicable.

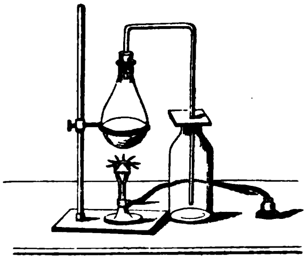
Hydrochloric acid gas and ammonia may be prepared to illustrate the process.

Preparation of Hydrochloric Acid Gas (HCl).[1]

48. This gas is easily obtained by the action of sulphuric acid on common salt.

To the neck of a dry Florence flask, a bent tube of the form represented in Fig. 14, is adapted by means of a perforated cork; and the flask is charged by introducing 300 grains of dry chloride of sodium (NaCl), and adding to it six fluid drachms of strong sulphuric acid (HO,SO_3). Immediate effervescence takes place, and the bent tube is passed down to the bottom of a dry bottle of about a pint capacity, which should be furnished with a greased stopper or glass plate; while the bottle is filling, the mouth may be loosely closed with a piece of card or paper.

Fig. 14.



Preparation of Hydrochloric Acid Gas.

49. Observe the dense fumes which are formed wherever the gas mixes with the air, especially if the atmosphere is damp, owing to the combination of the gas with the aqueous vapor. The bottle may be considered full when the gas has been flowing over from the mouth of the bottle for two or three minutes; the tube should then be slowly withdrawn, and the bottle tightly closed with the stopper or plate. Three or four bottles may be similarly filled with the gas, a gentle heat being applid when the evolution of gas slackens.

50. The decomposition may be thus represented:—

$$\mathrm{NaCl}+HO,SO_3=\mathrm{NaO,SO_3}+\mathrm{HCl}.$$

The sulphate of soda of course remains in the flask.

51. Ascertain the action of the gas on a piece of blue litmus paper.

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[1] The specific gravity of hydrochloric acid gas is 1.247, 100 cubic inches weighing 39.64 grains. Its atomic weight is 36.5, and its combining volume 4.

52. Remove the stopper from one of the bottles, instantly close it again with a dry glass plate (a precaution which is on no account to be omitted, as the stopper might in that case become immovably fixed), and plunge it with the mouth downwards into the water of the pneumatic trough. If the bottle has been well filled, the water will, when the glass plate is removed, quickly rise and nearly fill it, while the unabsorbed residue shows the quantity of common air left in the bottle.[1]

This experiment must not be made without first removing the stopper, and substituting the glass plate; if it is attempted to take out the stopper while the bottle is under water, there is great danger of its becoming so firmly fixed, as to be almost incapable of removal, owing to the absorption of the gas by the water, and the formation of a partial vacuum.

53. Test a little of the acid solution obtained in the last experiment, in a tube, with litmus paper, and afterwards with a few drops of solution of nitrate of silver (AgO,NO_5). The white precipitate, which is chloride of silver ($AgCl$), will be found to be insoluble in nitric acid, but readily soluble in ammonia.

54. Reserve a bottle of the gas for an experiment (60) with ammonia.

SECTION VIII.

Preparation of Ammoniacal Gas (NH_3).[2]

55. This gas may be collected in a similar manner to the last; but as it is specifically lighter than common air, the bottles in which it is collected must be kept, while filling, with the mouth downwards, the delivering tube passing upwards to the top (Fig. 15).

56. Mix 300 grains of powdered quick-lime with 300

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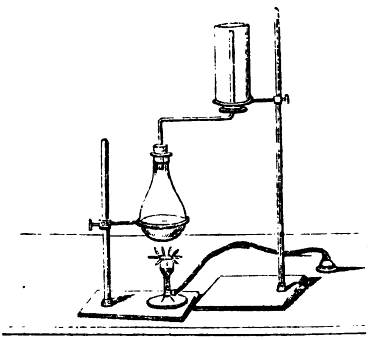
[1] **Water at common temperature is capable of dissolving no less than 480 times its own volume of hydrochloric acid. The liquid hydrochloric or muriatic acid of commerce is a solution of the gas in water.**

[2] **The specific gravity of ammoniacal gas is 0.589, 100 cubic inches weighing 18.288 grains. Its atomic weight is 17, and its combining volume 4.**

grains of chloride of ammonium (NH_4Cl) in a mortar, transfer the mixture to the flask, add a drachm and a

Fig. 15.



Preparation of Ammoniacal Gas.

half of water, mix this with the powders by agitation, and apply a very moderate heat. The following decomposition takes place:—

$$NH_4Cl+CaO=CaCl+\mathit{HO}+NH_3.$$

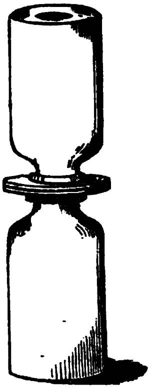
To ascertain when the bottle is filled with gas, hold a piece of moistened turmeric or red litmus paper near its mouth; a strong alkaline reaction will be perceived. When this is the case, slide a greased glass plate under the mouth of the bottle and slowly withdraw it, replacing it immediately by another. The closed bottle should be set aside with its mouth downwards. When three or four bottles have been filled, proceed with the following experiments:—

57. Observe the effect of the gas on a lighted taper slowly thrust up into the inverted bottle; it extinguishes the flame, and at the same time shows a slight tendency to burn with a pale green flame.

58. Remove the stopper from one of the bottles, and close the mouth with a dry glass plate (read paragraph 52); then invert it, and having placed it under water,

remove the glass plate and observe the rapid absorption. That which remains unabsorbed is atmospheric air.[1]

Fig. 16.



59. Test the liquid obtained in the last experiment (which is a weak solution of ammonia) with turmeric and reddened litmus paper; the first is turned brown, the latter has the blue color restored.

60. Remove the stopper from a bottle of the gas, and also from the reserved bottle of hydrochloric acid (54), replacing them with dry glass plates. Then invert the latter over the bottle of ammonia (Fig. 16), and cautiously remove the glass plates so as to allow the gases to mix. Dense white fumes, consisting of chloride of ammonium (NH_4Cl) are immediately produced, which in a short time collect in flakes, and fall like snow on the sides and bottom of the vessels. In this combination of the hydrochloric acid with the ammonia, considerable heat is evolved.

CHAPTER III.

DISTILLATION.

SECTION I.

Distillation of Water.

61. Adapt a cork to the neck of a quilled receiver, and bore a hole through it to fit the neck of the retort, which should pass through it for about two inches. When this is done, the apparatus may be fitted up as shown in the

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[1] **Water at common temperatures is capable of absorbing nearly 700 times its volume of ammoniacal gas.**

figure (Fig. 17). The funnel which supplies water for cooling the neck of the retort, has its throat partially obstructed by a plug of tow, so that the water may flow only drop by drop; the neck of the retort is covered by a slip of bibulous paper of the form of the annexed sketch (Fig. 18), cut of such a width as almost completely to encircle the neck, and tenderly laid on after being wetted; between the lower end of the paper and the quill receiver a thin fillet of wet tow is twisted tightly round the glass, to carry off the superfluous water, which drops into a basin placed underneath for its reception. The quill of the receiver passes into a small flask or bottle, which is kept immersed in water during the process, in order to keep it cool.

Fig. 17.

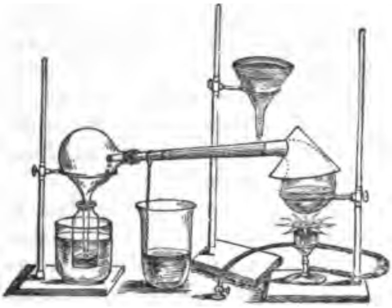
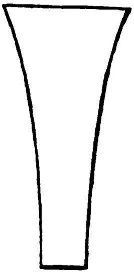


Fig. 18.

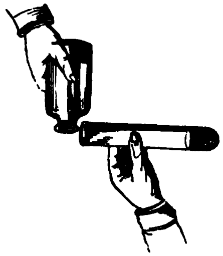


62. When the apparatus is thus arranged, the retort must be cautiously charged with common water till nearly half full, care being taken that none of it gets into the neck, as it would run down into the receiver and contaminate the distilled water, which should otherwise be pure. The upper part of the body of the retort being then covered with a conical cap of stiff brown paper to prevent loss of heat by currents of air and radiation, the lamp may be applied, care being taken that the ebullition

does not go on too violently, lest any of the impure water should splash or boil over into the neck of the retort. The first ounce of water that comes over should be rejected as impure, after which two or three ounces may be distilled for examination.[1]

63. While the ebullition is going on, another portion of the water operated on may be tested, with the view of discovering some of the impurities present in it. Fill four test-tubes about one-third full of the undistilled water, and add to them respectively a few drops of the following reagents:—

Fig. 19.



(*a.*) To the first add a solution of *chloride of Barium* ($BaCl$); a white precipitate, insoluble in dilute hydrochloric acid,[2] indicates the presence of sulphates, most commonly sulphate of lime (CaO, SO_3).

(*b.*) To another portion add a solution of *nitrate of silver* (AgO, NO_5). If any chloride is present (usually chloride of sodium ($NaCl$)), a white curdy precipitate of chloride of silver ($AgCl$) will be produced, insoluble in nitric acid, but readily soluble in ammonia. By exposure to the light this precipitate gradually becomes purple, especially when the water contains organic matter.

(*c.*) To the third tube add a little *lime-water* (CaO in water); a white precipitate, soluble in hydrochloric acid, shows that carbonic acid (CO_2) is present.[3]

(*d.*) To the remaining tube *oxalate of ammonia* ($NH_4O,$

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[1] A retort must never be heated before the liquid is introduced, and the flame must be so regulated during the distillation, that the sides of the retort above the level of the liquid may not be too highly heated.

[2] In testing the solubility of a precipitate in any liquid, pour off a small portion into a separate tube for the experiment, reserving the rest for comparison.

[3] This test would only detect carbonic acid if present in considerable quantity: its failure must not be assumed to indicate the entire absence of carbonic acid.

C_2O_3) may be added, which will give a white precipitate if any lime is present.

64. Test the distilled water in the same way; if pure, it will of course furnish no precipitate with any of the reagents.

65. Evaporate a few drops both of the distilled and undistilled water on platinum foil or a clean slip of glass; a considerable residue will probably be left by the latter, but no trace of solid matter ought to be observable where the other lay.

66. During ebullition, the water in the retort usually becomes turbid, owing to the formation of a white insoluble powder, which may be separated by filtration when the distillation is over.

To prepare a filter, take a small piece of white filtering or blotting paper, and fold twice from side to side (Fig. 20); then round off with scissors the projecting corners, so that the paper may fall wholly within the funnel (Fig. 21). Moisten the paper placed in a funnel with distilled water, and then carefully pour in the liquid to be filtered, using a glass rod to conduct it (Fig. 22).

Fig. 20.

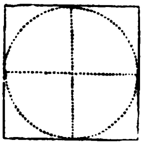
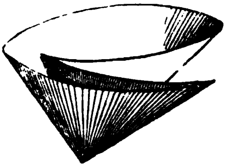


Fig. 21.

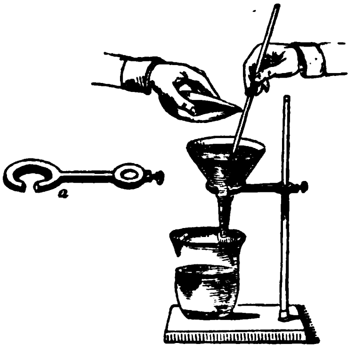


When the liquid has passed through, the filter and funnel are placed over another test glass; the clear solution being reserved for examination (68).

67. (*a.*) Pour a few drops of dilute *hydrochloric acid* over the powder upon the filter, and observe that it dissolves with effervescence, indicating that it is a carbonate.

(*b.*) Supersaturate the solution thus obtained with ammonia, and add a little *oxalate of ammonia* (NH_4O, C_2O_3); a white precipitate shows the presence of lime. The powder is thus proved to be carbonate of lime (CaO, CO_2). This carbonate of lime had been held in solution by the excess of carbonic acid contained in the water; when the gas is expelled during ebullition, the carbonate is precipitated.

Fig. 22.



68. Test the solution filtered from the carbonate of lime in (66) with *chloride of barium*, *nitrate of silver*, *lime-water*, and *oxalate of ammonia;* and compare the results with those obtained in (63), when the water was examined in its natural state. As most of the lime has been separated as carbonate, we may expect to find less of it in solution than before, but more of the sulphates and chlorides, since they still remain dissolved in a more concentrated form.

SECTION II.

Distillation of Liquid Hydrochloric Acid (HCl in water).

69. Fit up the apparatus as in the ordinary process of distilling water (61), taking care that the cork is perfectly air-tight; then remove the retort, and introduce through the tubulure 1000 grains[1] of dry chloride of sodium ($NaCl$)

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[1] In this and many other of the experiments, small quantities are mentioned to suit the convenience of my class of Practical Chemistry, the lessons being only two hours long. When the products of the experiments are wanted for use, much larger quantities must frequently be employed.

in coarse powder, taking care that none of the particles fall into the neck of the retort; then adjust the apparatus as before. Measure into the small flask or bottle which is to receive the distilled acid, 12 fluid drachms of water, and mark with a file or a strip of waxed paper, the height at which it stands; and having emptied it, measure into it seven drachms of distilled water, which will be increased to 12 drachms by the hydrochloric acid which distils over. During the distillation care must be taken that the quill of the receiver dips just under the surface of this water, which will assist in condensing the acid fumes, some of which might otherwise escape; but the tube need not pass to the bottom of the water, because the solution of hydrochloric acid is the heavier of the two.

Into a small evaporating basin pour seven drachms of water, and add gradually to it six drachms of strong sulphuric acid (HO,SO_3) stirring the mixture with a glass rod. When nearly cool, this dilute acid may be poured carefully into the retort through a small funnel, avoiding any splashing or soiling of the neck. A gentle heat may then be applied, which must be regulated according to the rapidity with which the acid distils over, great care being taken that the mixture does not boil over into the neck of the retort (50).

The distillation may be continued until the liquid in the receiving bottle has increased to twelve drachms of acid, which may be known by the mark previously made.

70. The acid in the receiver may now be examined as to its purity. Pour a little into a test-tube, dilute it with about three times its bulk of water, and add a few drops of a solution of *chloride of barium;* if a white precipitate appears which is insoluble in the acid, it shows the presence of sulphuric acid as an impurity.

71. Evaporate a few drops of the acid on platinum foil or a clean strip of glass; no trace of the spot where it lay ought to remain. Any solid residue shows the presence of some saline impurity, caused probably by a little of the salt employed having got into the neck of the retort, and been washed down into the receiver.

SECTION III.

Distillation of Liquid Ammonia (NH_3 in water).

72. Prepare the apparatus as in the distillation of hydrochloric acid (69).

Pound 450 grains of quick-lime (CaO), introduce it into the retort through the tubulure, and pour gradually upon it two ounces of distilled water. Measure into the receiving flask or bottle fifteen drachms of water, and mark with a file or waxed paper the height at which it stands; empty it, and pour in two drachms of distilled water for the quill of the receiver to dip into during the distillation.

Weigh out 530 grains of chloride of ammonium (NH_4Cl), dissolve it in three ounces of water in a small evaporating basin, and pour the solution into the retort.

The distillation may now be commenced, carefully regulating the heat, and continuing it until the distilled liquid reaches up to the file mark of the receiver, when 15 drachms will have been obtained.

$$CaO + NH_4Cl = NH_3 + HO + CaCl.$$

73. Pour a little of the ammoniacal solution thus prepared into a test-tube, and add to it a few drops of *chloride of barium;* if a precipitate appears, it is owing to the presence either of carbonic or sulphuric acid. To distinguish between them, add dilute *hydrochloric acid* in slight excess; if the precipitate thereupon dissolves, it is due to carbonic acid; if not to sulphuric.

74. Test another portion of the ammoniacal solution with a little *oxalate of ammonia;* if a white precipitate is formed, it is owing to the presence of lime as an impurity.

75. Supersaturate a little of the distilled liquid with nitric acid in a test-tube, and add a few drops of a solution of *nitrate of silver* (AgO,NO_5); a white precipitate indicates the presence of hydrochloric acid or a chloride. If a further portion of the ammoniacal solution be added, so as to render the liquid alkaline, the precipitate redissolves.

76. If no precipitate occur with any of these tests,

evaporate a few drops of the ammoniacal solution on a slip of glass or platinum foil, and observe whether any trace of saline impurity is left.

SECTION IV.

Distillation of Liquid Nitric Acid (HO,NO_5 in water).

77. Fit up the apparatus as in the distillation of hydrochloric acid (69).[1] Introduce into the retort 1000 grains of nitrate of potash (KO,NO_5); pour upon it ten drachms of strong sulphuric acid (HO,SO_3) previously diluted with an equal bulk of water, and apply a gentle heat, observing the same precautions as were recommended in the former cases (61, 69).

$$KO,NO_5+2(HO,SO_3)=KO,SO_3,HO,SO_3+NO,HO_5.$$

78. While the distillation is going on, dissolve a few crystals of the nitrate of potash in distilled water, for the purpose of ascertaining its purity.

(*a.*) Test a little of the solution with *nitrate of silver* (AgO,NO_5); if any chloride is present, a white curdy precipitate appears, which is insoluble in nitric acid, but readily soluble in ammonia.

If the nitrate employed is contaminated with any chloride, the acid that distils over is sure to contain a little chlorine.

(*b.*) To another portion, add a solution of *chloride of barium* ($BaCl$); if any sulphates are present, a white precipitate is produced, which is insoluble in dilute hydrochloric acid.

79. Dissolve 250 grains of nitrate of potash in an ounce and a half of hot water, in an evaporating basin, and observe the gradual formation of crystals as the solution cools. Remove some of these from the liquid, and dry them on filtering paper; then redissolve them in distilled water, and test the solution as before, with *nitrate of silver* and *chloride of barium*. The precipitates, if any, will

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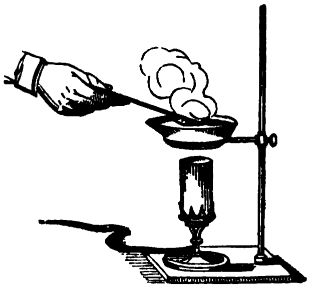
[1] In preparing this acid upon a larger scale, it would be necessary to avoid the use of cork, which is acted upon by the acid; the neck of the retort would then be merely slipped into that of the receiver.

be less dense than in the previous examination, showing that a partial purification has been effected.

Fig. 23.



80. The distilled nitric acid may now be tested for impurities, but before the test liquids are applied, a portion should be diluted with four or five times its bulk of distilled water, since the chloride of barium and nitrate of baryta are insoluble in strong nitric acid, and would consequently cause a precipitate even though no sulphuric acid were present. A portion may then be tested for sulphates and chlorides with *chloride of barium* and *nitrate of silver*.

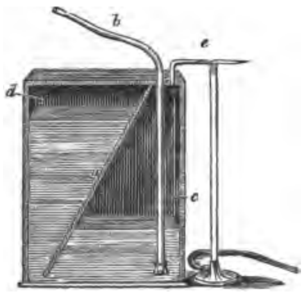
CHAPTER IV.

GLASS-WORKING.

81. The most convenient form of apparatus for working glass on the small scale, is the water blowpipe, which consists of an upright box, about fifteen inches high, of the form represented in Figure 24. It is usually made of zinc or copper, and is divided into two compartments by the plate *a*, which passes down to within about half an inch of the bottom, thus leaving a communication open between the two. The lower end of the tube *b* is closed by a valve opening outwards, to prevent the escape of air in that direction; the box should be filled about half full of water, and when used, air is blown through the tube *b*. The pressure thus occasioned in the compartment *c*, forces a portion of the water into the next division *d*, where it rises to a higher level than in *c*, and by its superior pressure forces a stream of air through the fine aper-

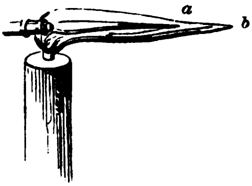
ture at the extremity of the tube *e*, as long as it continues to stand at a higher level than at *c*. In this way a continuous jet is readily obtained, with much less fatigue to the operator than with the mouth blowpipe.

Fig. 24.



Water Blowpipe.

Fig. 25.



Blowpipe Flame.

82. If the blowpipe flame be examined, it will be found to consist of two distinct parts, which may be called, for the sake of distinction, the inner *a*, and the outer flame *b* (Fig. 25).[1] The blue point of the inner flame is evidently surrounded on all sides by the burning gas, the oxygen of the air projected into the flame having been entirely consumed before this point of the flame is reached, so that any substance containing oxygen loosely combined, placed in it, will be decomposed by the powerful deoxidizing affinities of the carbon and hydrogen of the combustible gases; on this account the inner flame is usually called the *deoxidizing* or *reducing* flame. The outer flame, on the contrary, is surrounded on all sides by the external air, so that here there is no excess of combustible or deoxidizing matter, but rather an excess of atmospheric oxygen; so that an oxidized substance may be placed at its extremity without danger of deoxidation, unless such decomposition is effected by the mere heat of the flame, independent of its chemical action; on the other hand, most substances, having an affinity for oxygen, placed within its influence, become oxidized at high tempera-

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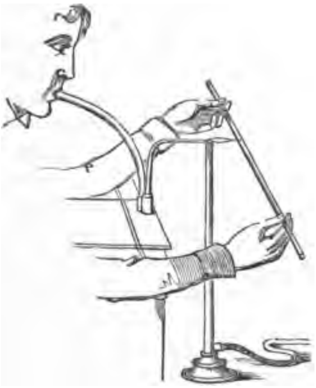
[1] Three parts will really be observed; but the innermost dark portion is merely a hollow space filled with the comparatively cool mixture of combustible gas and air.

tures, and hence it is usually called the *oxidizing* flame. The hottest part of the blowpipe flame is a very little in advance of the point of the inner blue cone (reducing flame).

83. The English flint glass, of which the tubes and rods commonly in use are made, contains in its composition a quantity of oxide of lead (PbO), which, when heated in contact with deoxidizing matter, is very easily decomposed. On this account it is necessary, in heating such glass with the blowpipe, to take care that it does not approach the deoxidizing flame, but is kept at the extremity of the oxidizing flame, otherwise a black stain of metallic lead will be deposited on the surface of the glass. Slight stains of this description may generally be removed by holding the glass for a few seconds in the oxidizing flame; this converts the lead again into oxide, which dissolves in the glass. The German hard glass tubing, which is free from lead, and is much less fusible than the English glass, is far preferable for making tubes, &c., which will be required to withstand a high temperature.

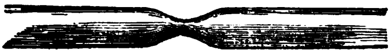
Fig. 26.



84. *Make a few glass stirring rods, of lengths varying from five to eight inches.* To do this, a piece of solid rod, long enough to make two stirrers, should be held at a short distance from the extremity of the flame, and gradually brought towards it; a rotating motion being communicated to it by means of the finger and thumb, so that the part where the heat is applied may be uniformly heated all round (Fig. 26). When the glass begins to soften, it should be gently pulled with both hands, until it assumes the form represented in Fig. 27, when it may be removed from the flame; and having been

scratched with a file across its narrowest part, is gently broken asunder (Fig. 28).

Fig. 27.



85. The sharp edges are then held in the flame until they are rounded, so as to form a blunt conical termination to the rod, whilst the other extremity may be simply fused till it is round and uniform (Fig. 29).

Fig. 28.

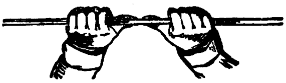
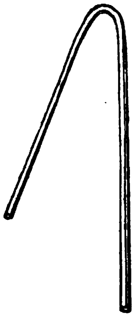


Fig. 29.

86. *Make a small syphon tube.* Take a piece of tubing ten or twelve inches long, and a fourth or a third of an inch in diameter, and hold it diagonally in the upper part of the flaring flame of a gas or spirit lamp (*not in the blowpipe flame*), turning it constantly round, and by gently moving it up and down in the flame, heating two or three inches of the central part of the tube. When the glass begins to soften, apply a gentle pressure with both hands, so as to bend it slowly, and continue to do so until it has assumed the form shown in Fig. 30. If the tube be too strongly heated, or the pressure too strongly and suddenly applied, the bend, instead of being round and uniform, will be abrupt and wrinkled, in which case it is very liable to crack, either spontaneously, or when exposed to slight variations of temperature. The extremities of the tube must now be rounded off by being heated to redness for a moment in

Fig. 30.



Syphon Tube.

the flame of the blowpipe. When the glass operated on is at all thick, or of an unequal form, some care is necessary in *annealing*, or gradually cooling it; this may be effected by removing it slowly from the flame, and then laying it across a piece of tube, so that the hot part does not touch any cold substance, and covering it loosely with paper, to prevent too rapid cooling by radiation.

87. *Make a few test-tubes.* A piece of tube may be taken about half or five-eighths of an inch in diameter, and eight or ten inches long, which will serve for two test-tubes.

Fig. 31.

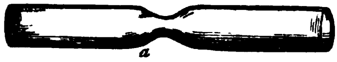
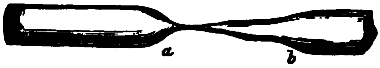


Fig. 32.



Making Test-tubes.

In cutting off a piece of wide thin tubing such as is employed for test-tubes, it is advisable to carry the file-mark round the tube, which is then to be held in a cloth and broken by a combined pulling and breaking movement of the hands. If the end of the broken tube exhibit a crack, this must be led round the tube by applying a red-hot iron point (the end of a file heated by the blowpipe flame will do) a little in advance of it.

The central portion must be heated in the manner described for heating glass rod (84), and gradually drawn out, the tube being constantly turned round, when it will assume the form shown in Fig. 31. The heat should now be applied to the part of the tube marked *a*, and the other piece gradually drawn out, care being taken not to fuse the thin thread of glass which is formed, and which connects the two parts of the tube (Fig. 32), until the base of the tube has become round and uniform; when this is the case, and the connecting thread has become very thin, the heat may be applied to the point where it joins the tube, when it will instantly fuse and separate, leaving the tube in an almost finished state. There will generally be found at the bottom of the tube, however a small lump, more or less distinct, formed by a portion of the thread having fused into it; to remove this, again heat the round end red-hot for a short time, until the lump disappears. On removing the tube from the flame, blow air gently into it, for the purpose of swelling out the bottom to its previous round form (Fig. 33), as it usually collapses and flattens while in a state of fusion.

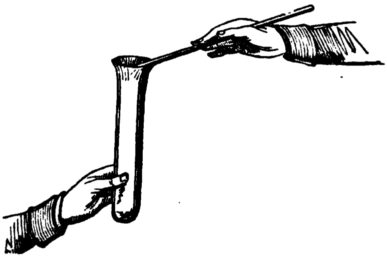
Fig. 33.

88. The other portion of the tube may now be finished in a similar way, by applying the heat to the point *b*, and drawing off the irregular termination until the thread of glass is sufficiently attenuated to be removed.

When it is required to make a test-tube of a piece of tubing only long enough for one, all that is necessary is to melt on to one end another piece of waste tubing or rod, to serve as a handle, after which the end may be drawn off, as in the former case.

89. To complete the tube the open end must be spread out a little, as shown in Fig. 34, so as to form a kind of border. This is done by softening the end in the blowpipe flame, and then, by means of a thick iron wire, or the smooth end of a file, introduced and carried round the opening, the edge is uniformly pressed outwards.

Fig. 34.

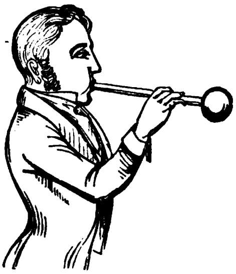


Making Test-tubes.

90. *Blow some small bulbs.* When it is required to blow a bulb at the end of a tube, the extremity should be closed as in making a test-tube (87); if the glass is tolerably thick, and the bulb to be blown not large, all that is necessary is to heat the end for about half an

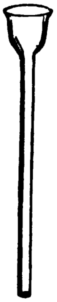
inch as strongly and uniformly as possible; and then, having removed it from the flame, and holding it horizontally in the hands (Fig. 35), to blow air into it until the pressure forces the softened glass to expand, which it will do in the form of a round bulb if the heat has been properly applied, and the tube be kept constantly turned round while in the hands. This latter precaution is absolutely necessary, as the softened glass would otherwise bend with its own weight in one direction, thus destroying the proper form of the tube.

Fig. 35.



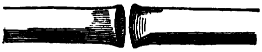
91. *Make a funnel tube.* Blow a bulb of considerable size on the extremity of a piece of strong tube about a quarter of an inch in diameter. Heat a spot upon the bulb immediately opposite to the point at which the tube enters it, and blow suddenly into it until it bursts; break away the superfluous thin glass with the fingers, and finish off the border with the blowpipe flame and the heated end of a file.

Fig. 36.



Funnel Tube.

Fig. 37.



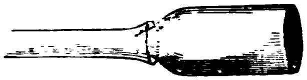
Tubes united.

92. *Cement together two tubes of equal diameter.* The ends to be joined must be cut perfectly level; the edges are brought exactly together, the tubes being kept in the same straight line, and the glass is gradually softened by rotating the tube slowly in the outer flame; a gentle pressure may be applied to perfect the joint, and a little care in moulding it will soon render it perfectly uniform; it should be well annealed by withdrawing it slowly from the flame. When the glass is thin, the edges which are to be united may be spread out a little,

as shown in Fig. 37, by means of a heated wire or file (89), when the joint will be stronger than it would otherwise be.

93. *Cement together two tubes of unequal diameter.* When it is required to join a narrow tube to a wider one, it is necessary to draw out the latter in the blowpipe flame until a portion of it is contracted to the diameter of the former (87); then with a file it is divided at that point of equal diameter, and cemented to the smaller tube in the same way as in the previous case. Sometimes, when the glass is thin, it is advisable to widen the extremity of the smaller tube, so as to overlap the other (Fig. 38), which is readily done by means of an iron wire (89).

Fig. 38.

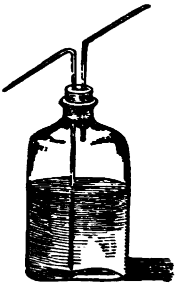


Tubes united.

In this operation, it is always advisable to maintain the junction in the flame for some little time, to allow of the complete amalgamation of the two portions of glass; and as the tendency to collapse is greater the longer it is fused, it will generally be found necessary to blow it out slightly.

94. *Prepare tubes for a washing bottle.* The tubes required for this purpose are of the form shown in Fig. 39, the upper end of the longer one being drawn out so as to leave only a small aperture.

Fig. 39.



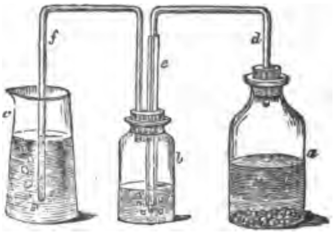
Washing Bottle.

When the bottle is prepared and filled with water, a small stream of water may be forced through this tube by blowing air down the shorter one: it is of great service in washing precipitates on a filter, and for many other purposes.

95. *Prepare tubes for a sulphuretted hydrogen (hydrosulphuric acid) apparatus.* The form of apparatus used for

generating hydrosulphuric acid, and passing it into water or saline solutions, is represented in Fig. 40. The finer tubes, *d* and *f*, are bent in the naked flame (86), the extremities being afterwards slightly fused with the blowpipe in order to round off the sharp edges; and care must be taken that the wider tube *e* is of sufficient calibre to admit of the tube *d* passing freely down it.

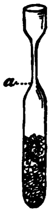
Fig. 40.



Sulphuretted-hydrogen Apparatus.

96. *Seal a few tubes hermetically at both ends.* This is an operation of very frequent use in the laboratory, as it furnishes the most convenient and efficient means of preserving small specimens of many rare substances, especially such as are volatile, and is often required in effecting chemical changes at elevated temperatures and pressures.

Fig. 41.



The tube is first sealed at one end, precisely as if it were intended for a test-tube (87); the liquid or other substance for which it is designed is then introduced, as soon as the tube is quite cold, care being taken that the upper part of the tube is not wetted or soiled. The flame of the blow-pipe is now directed to the portion of the tube a little above that intended for the sealed end, and when sufficiently soft it is drawn out to a capillary tube, and allowed to cool; it may afterwards be sealed by fusing the lower part of the capillary tube *a* (Fig. 41), by momentary contact with the flame.[1]

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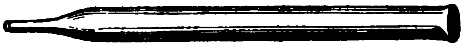
[1] When the tube is required to withstand high pressures, the sealing of both ends must be effected very slowly, so that the glass may remain as thick there as at any other part of the tube.

In this way seal a little sulphur in a tube without melting or volatilizing any of it, the sulphur being within an inch and a half of the upper end.

97. *Seal some water hermetically in a tube.* Having sealed the tube at one end, and drawn it partly out at the other, leaving a rather wide neck, while it is cooling take another piece of tubing, which may be eight or nine inches long, and a sixth of an inch in diameter, and draw it slowly out in the blowpipe flame; then divide it in the thin part by means of a file, when it will have the form shown in Fig. 42; and when the sharp edges have been rounded off in the blowpipe flame, may be used as a pipette for introducing a little water into the sealed tube without wetting its sides.

Fig. 42.



Then draw out the capillary neck, and when cold, seal it as before, leaving not more than the space of an inch between the upper end and the surface of the water.

CHAPTER V.

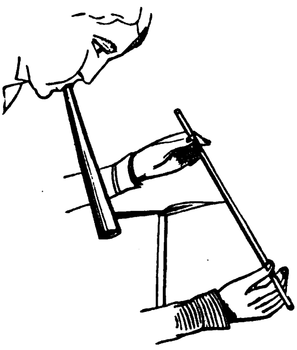
EXPERIMENTS WITH THE MOUTH BLOWPIPE.

98. Before proceeding to any blowpipe experiments, it is necessary to acquire the power of keeping up a constant and unintermitting blast of air from the mouth, as without this it is impossible to raise the heat to a sufficient degree of intensity. The habit is readily acquired, and when once attained, the mouth and lungs will be found to do their work almost mechanically, without any sustained effort on the part of the operator.

The learner must first observe that on closing the lips he can still without any difficulty breathe through the nostrils; let him now distend the cheeks with air, and he will find that he can breathe through the nostrils for a length of time, still keeping the cheeks distended. He may next introduce the mouth-piece of the blowpipe between his lips, and having puffed out his cheeks with air, let him breathe freely through the nostrils, at the same time allowing the distended cheeks to force a current of air through the blowpipe. The cheeks thus play the part of an elastic bag, which, if connected with the blowpipe, and distended with air, would force air through it as long as the tension of its stretched sides exerted sufficient pressure.

99. Seal a few tubes for the following experiments (Fig. 43). The tubing employed for this purpose should be about a quarter of an inch in diameter, and it may be scratched with a file, and broken into pieces about four inches long, each of which will serve for two tubes. The glass of which it is made should not contain lead. The sealing should be effected in the manner already described (87). It is not necessary to be particular about rounding off the ends of these tubes, but they should be carefully annealed by withdrawing them gradually from the flame. In using one of these tubes for an experiment, it should first be heated in the common flame, the blowpipe flame being afterwards applied, if the former does not give enough heat; the tube frequently cracks if the blowpipe flame be suddenly applied.

Fig. 43.



These tubes may be most conveniently held by a narrow strap of folded paper passed round the upper part.

Very small quantities should be used in the following experiments. A fresh tube must be employed in each.

100. Heat a little crystallized sulphate of iron (copperas

or green vitriol, FeO,SO_3+7Aq) in a tube,[1] without using the blowpipe; observe that the salt loses its green color, water being expelled and condensed in drops upon the upper part of the tube, which should be held almost horizontal, lest the water run down to the heated glass, and crack it. On applying the blowpipe flame to the bottom of the tube, the sulphuric acid will be expelled, and red peroxide of iron left.

$$2(FeO,SO_3)=SO_3+SO_2+Fe_2O_3.$$

The odor of sulphurous acid may be perceived at the mouth of the tube.

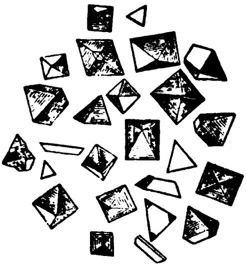
Test the drops of water with a piece of blue litmus paper inserted into the tube; they will be found strongly acid.

101. Heat a little phosphate of soda and ammonia (microcosmic salt, NaO,NH_4O,HO,PO_5+8Aq) in a similar manner; test the condensed water with red litmus paper, to show that it is strongly alkaline from ammonia, which may be recognized by its odor. Observe that the residue finally fuses into a transparent glass which is metaphosphate of soda (NaO,PO_5).

102. Heat a little sulphur in a tube; notice its easy fusion, its darkening in color, its distillation in yellow drops upon the cool part of the tube, its peculiar odor at the mouth of the tube.

103. Heat a minute quantity of arsenious acid (white arsenic, AsO_3), taking the precaution to warm the upper part of the tube by drawing it once or twice through the flame before heating the arsenious acid; the vapor of the latter will then condense slowly upon the glass, forming a *sublimate* of minute shining crystals, which appear as octohedra (Fig. 44) when magnified.

Fig. 44.



Arsenious Acid.

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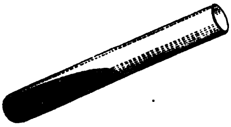
[1] If a gas flame be employed, the tube must be held in the lower blue portion, so as not to be smoked.

104. Heat a little calomel (Hg_2Cl) and corrosive sublimate ($HgCl$) in separate tubes; observe that the former sublimes without fusing, whilst the latter fuses to a colorless liquid, and afterwards sublimes. The fused corrosive sublimate solidifies in beautiful needles on cooling. These may often be seen projecting from the sides, on looking down the tube.

105. Dry a little powdered carbonate of soda (NaO,CO_2) (upon a strip of tin plate) at a moderate heat, and mix with a small portion of it a very minute quantity of calomel (not more than a tenth part of the weight of the carbonate of soda); introduce the mixture into a tube, cover it with a little more carbonate of soda, cleanse the upper part of the tube with a roll of paper, shake the mixture a little to one side of the tube (Fig. 45), to allow the passage of the vapor, and apply heat first to the layer of carbonate of soda, then to the mixture, finally using the blowpipe. Observe the deposition of a gray crust composed of minute globules of mercury on the cool part of the tube. The globules will become more evident on rubbing the crust with a slender glass rod.[1]

Fig. 45.

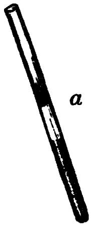


Reduction of Calomel.

106. Scrape a little powder from a piece of charcoal, mix it with three times as much dry carbonate of soda, add to the mixture a very minute quantity of arsenious acid (not more than a tenth part of the weight of the mixture), and proceed as in the last experiment. A shining black crust of metallic arsenic will be formed upon the upper part of the tube (Fig. 46). An odor of garlic will probably be perceived at the orifice.

Cut off, with a file, the portion of the tube upon which the deposit has formed, wrap it in a piece of paper, break it into small fragments, and heat these gently in another tube,

Fig. 46.



Crust of Arsenic.

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[1] Easily made by drawing out a piece of glass rod in the blowpipe flame.

the upper part of which has been first warmed. The oxygen of the air in the tube will convert the arsenic into arsenious acid, condensing in brilliant octohedra on the upper part.

107. Mix together equal portions of nitre (KO,NO_5) and bisulphate of potash ($KO,HO,2SO_3$), and heat the mixture in a tube; test the red vapor which is given off with blue litmus paper, and endeavor to account for its formation.

108. Mix a little iodide of lead (PbI) with bisulphate of potash, and heat the mixture in a tube; the beautiful violet-colored vapor which rises and condenses in the upper part of the tube is iodine.

$$PbI+2(KO,HO,2SO_3)=PbO,SO_3+2(KO,SO_3)$$
$$+2HO+SO_2+I.$$

109. Heat a mixture of powdered fluor spar (CaF) and bisulphate of potash in a tube. Observe the remarkably pungent vapors of hydrofluoric acid (HF). When the tube is cool, cut off the upper part, well wash and dry it; the glass will be found dimmed and corroded by the action of the acid.

$$KO,HO,2SO_3+CaF=HF+CaO,SO_3+KO,SO_3.$$

110. Heat a small fragment of wood ($C_{12}H_{10}O_{10}$) in a tube; observe that it chars or blackens, like most organic substances, from the separation of carbon. Test the vapor with a strip of moistened blue litmus paper, which will be reddened by the acetic or pyroligneous acid ($HO,C_4H_3O_3$) resulting from the destructive distillation of the wood.

111. Treat a shred of isinglass (gelatine $C_{48}H_{39}N_7O_{17}$) in a similar way; compare the odor with that observed in the last experiment, and test the vapors with moistened red litmus paper, which will show them to be alkaline from the presence of ammonia arising from the decomposition. Nitrogenized organic matters usually evolve alkaline vapors having this peculiar odor.

112. Scoop a shallow cavity in a piece of charcoal, place a little nitrate of potash in it, and direct the blowpipe flame upon it; notice the *deflagration* due to the

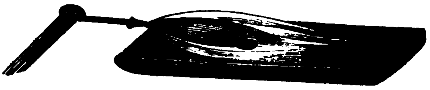
rapid combustion of the charcoal in the oxygen furnished by the nitre.

113. Heat a very minute quantity of arsenious acid on charcoal; observe the garlic odor attending the re-oxidation of the metal.

114. Heat a little chalk or marble (CaO,CO_2) on charcoal, and note the dazzling white light (*incandescence*) which it emits. During the ignition, the carbonate of lime is decomposed, and quick lime (CaO) is left, the alkaline nature of which may be shown by placing a particle of it upon moistened red litmus paper, which will be found not to be affected by the marble before ignition.

115. Place a little red oxide of lead (Pb_3O_4) in a shallow cavity scooped very near to one end of a piece of charcoal; cover the oxide with carbonate of soda, and direct the inner blowpipe flame upon it, allowing the outer flame to sweep over the surface of the charcoal (Fig. 47). A globule of metallic lead will be obtained, and a yellow incrustation of protoxide of lead (PbO) will be formed upon the charcoal under the oxidizing flame. Place the globule of metal upon a hard surface (a small anvil or an inverted mortar), and strike it with a hammer (or a pestle); it will be found very soft and malleable, flattening out to a thin plate, which will make a dark streak upon paper when drawn across it.

Fig. 47.



Reduction on Charcoal.

116. Reduce oxide of bismuth (BiO_3) in the same way; observe that it gives a yellow incrustation and a brittle globule of metal.

117. Fuse a little chloride of silver ($AgCl$) on charcoal with carbonate of soda; a brilliant white globule of silver will be obtained, which does not tarnish in the outer flame, and is malleable. There should be no incrustation upon the charcoal.

118. Repeat the experiment with binoxide of tin (SnO_2);[1] the globule of metal will be readily tarnished in the outer flame; it is malleable, and gives little or no incrustation on the charcoal.

119. Heat a little sulphide of antimony (SbS_3) (or any other compound of antimony) on charcoal in the outer blowpipe flame; observe the odor of sulphurous acid (SO_2), indicating the presence of sulphur; cover the specimen with carbonate of soda, and fuse in the inner flame. A very brittle bead of antimony will be obtained, which emits fumes of oxide immediately after its removal from the flame. A copious white incrustation covers the surface of the charcoal.

120. Reduce a small quantity of sulphate of copper (CuO,SO_3+5Aq) on charcoal with carbonate of soda, and fuse the metal to a globule in the focus of the flame (the point of a sharp blue inner flame). Hammer the globule to prove that it is metallic copper. There is no incrustation on the charcoal.

121. Fuse sulphate of zinc (ZnO,SO_3+7Aq) with carbonate of soda in a powerful reducing flame; the metal will be volatilized as fast as it is reduced, being deposited again as an incrustation of oxide which is yellow whilst hot, and becomes white on cooling.

122. Mix a little sulphate of baryta (BaO,SO_3), or any other sulphate with charcoal, and heat it on charcoal in the reducing flame.[2] When the mass is cool, place it upon a clean silver coin, and moisten it with a drop of dilute hydrochloric acid; observe the odor of hydrosulphuric acid (HS), and the black stain of sulphide of silver (AgS) upon the coin.

In this experiment the sulphate of baryta is deoxidized by the charcoal, becoming sulphide of barium (BaS), which, when acted on by hydrochloric acid, is decomposed, liberating hydrosulphuric acid.

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[1] It is far more easy to reduce tin with cyanide of potassium than with carbonate of soda. In this, and other cases, the addition of a little borax will be found to facilitate the accumulation of the metal into a single globule.

[2] For this and the following experiments where no incrustation is to be looked for, circular slices cut from sticks of charcoal may be used; they may be conveniently held in a loop of tin-plate, in the manner shown in Fig. 48.

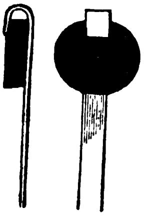
Decomposition during ignition.

$$BaO,SO_3+C_4=BaS+4CO.$$

Decomposition caused by the hydrochloric acid.

$$BaS+\mathit{HCl}=\mathit{BaCl}+HS.$$

Fig. 48.



Charcoal-holder.

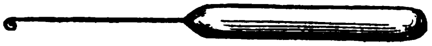
123. Ignite a little alum ($Al_2O_3,3SO_3,KO,SO_3+24Aq$) on charcoal, and moisten it with a drop or two of solution of nitrate of cobalt (CoO,NO_5); on again strongly igniting it, a beautiful blue mass will be obtained.[1]

124. Repeat the experiment with sulphate of zinc, which will give a fine green mass.

125. When sulphate of magnesia (MgO,SO_3+7Aq) is treated in the same way, it gives a very pale pink mass, the color of which becomes more distinct as it cools.

126. Fuse a glass handle to a piece of thin platinum wire, bend the end into a small loop, as shown in Fig. 49. Heat the loop in the inner blowpipe flame, when the outer flame will be colored yellow by the sodium derived from the fingers in making the loop. Continue to heat the wire till it no longer tinges the flame.[2]

Fig. 49.



127. Moisten the loop with hydrochloric acid, and take a little nitrate of potash upon it; on exposing this to the

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[1] This cannot be relied upon as a test for alumina, since some phosphates, &c., exhibit a similar reaction.

[2] The coloring power of sodium is so great, that it often masks the colors which other metals are capable of imparting to the flame, and hence this mode of testing loses much of its value in the presence of sodium.

inner flame, the outer flame will assume a transient violet tinge, due to the potassium.

128. Test a little marble in the same way; the orange red tint imparted to the outer flame indicates the presence of calcium. After the specimen has ceased to color the flame, it will regain the property when moistened with more hydrochloric acid.

129. Nitrate of strontia will color the outer flame of a beautiful crimson, caused by the metal strontium.

130. Sulphate of baryta, well moistened with hydrochloric acid, and exposed to a strong inner flame, colors the outer flame bright green, indicating barium.

131. Make another loop on the wire, rather larger than that used for the colored flames, heat it to redness, and dip it into some powdered borax ($NaO,2BO_3+10Aq$); heat it in the blowpipe flame, and observe that the borax *intumesces* and finally fuses to a colorless glass; whilst this is red-hot, take up a very minute particle of chromate of potash (KO,CrO_3), or any compound containing chromium, on it, and heat it again in the oxidizing flame; a yellowish-green transparent glass will be obtained, which will become pure emerald green in the reducing flame.

132. Carefully open the loop so as to remove the bead, and having made another bead of borax, test a little sulphate of copper in the same way; a blue glass will be formed in the outer flame, the color of which will disappear in the inner flame.

133. Dip a fresh bead of borax into solution of nitrate of cobalt, and fuse in the blowpipe flame: a beautiful blue glass will be obtained in both flames.

134. Binoxide of manganese (MnO_2) will impart to the borax glass an amethyst purple or violet color in the outer flame, which fades and finally disappears altogether in the inner flame.

135. Sulphate of iron, or any other compound of that metal, colors the glass brownish yellow in the outer flame, the color fading as it cools, and becoming bottle-green in the reducing flame.

136. Sulphate of nickel (NiO,SO_3) imparts to the glass a brownish-yellow color in the outer, and a dusky-gray in the inner flame.

137. Moisten the loop of platinum wire, and dip it into powdered carbonate of soda; fuse this into a bead, and while it is red-hot, take a very minute particle of black oxide of manganese upon it; fuse it again in the oxidizing flame, and observe that the opaque bead is green while hot, and blue on cooling; heat it in the reducing flame, and the color disappears.

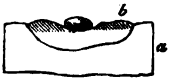
138. Make another bead of carbonate of soda, and observe that it becomes opaque on cooling. Heat it again to redness, dip it into powdered silica (SiO_2) and fuse it; observe the effervescence due to the expulsion of carbonic acid; after repeated fusions with silica, the bead will be found to remain transparent after cooling.

138*a*. Make a bead of microcosmic salt (NaO,NH_4O,HO,PO_5+8Aq), and fuse a little silica with it; observe that the silica floats about undissolved in the hot bead, as a semi-transparent mass, becoming more visible when the bead cools.

139. Mix a little borax with some fluor spar and bisulphate of potash; take a little of the mixture upon a clean platinum loop, and heat it in the inner blowpipe flame, when the outer flame will be tinged green, indicating the presence of boracic acid.

140. Scoop out a large shallow cavity in a piece of charcoal, *a* (Fig. 50), and fill it with powdered bone-ash (phosphate of lime, $3CaO,PO_5$) rammed very tightly down (*b*); place a fragment of argentiferous lead upon it, and expose it to the oxidizing flame; observe that the oxide of lead (PbO), as it is formed, fuses and is absorbed by the porous phosphate of lime. Continue the experiment until a small bead of silver is left, which is no longer tarnished in the outer flame. It may be found necessary to remove the metal to a fresh bed of bone-ash. This process is called *cupellation*.

Fig. 50.



The minute bead of silver may be identified as in (342).

Systematic examination of unknown substances with the blowpipe.[1]

141. Heat the powdered substance in a small closed tube of hard glass (100).

(*a*) Is any *water* condensed on the cool part of the tube? Is the water **acid** or **alkaline** to test-papers?

If acid, the presence of *sulphuric*, *hydrochloric*, or *nitric acid* may be suspected.

If alkaline, *ammonia* is probably present (101).

(*b*) Is there any **sublimate** deposited on the upper part of the tube?

This may be due to the presence of *sulphur* (102), of *arsenic* (103), of *mercury* (104), or of *ammonium.*

(*c*) Does the substance **carbonize** and emit any peculiar odor?

Indicating the presence of *organic matter*, either nitrogenized (111), or non-nitrogenized (110).

142. Mix a little of the powdered substance with dry carbonate of soda and charcoal and heat in a tube to detect *ammonium* (by the odor and alkaline vapors), *mercury* (105), and *arsenic* (106).

143. Heat the substance in a tube with bisulphate of potash.

Brown acid vapors indicate *nitric acid.*

Violet vapors indicate *iodine.*

Yellow bleaching vapors indicate *chlorine* (either as a chloride, a hypochlorite, or a chlorate).

Colorless pungent acid vapors indicate chlorine as a *chloride*, or fluorine as a *fluoride* (109).

144. Heat a little of the substance on charcoal.

Deflagration indicates a *nitrate* or *chlorate.*

Odor of sulphurous acid indicates *sulphur*, a *sulphide*, or a *sulphate.*

Odor of garlic indicates *arsenic.*

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[1] A list of substances which may be examined as exercises in this systematic course will be found in the Appendix.

White incandescent mass. Moisten with nitrate of cobalt, and heat again, to detect *zinc* (green), *aluminum* (blue), or *magnesium* (pink). (*See* 123, note.)

145. Fuse the substance with carbonate of soda on charcoal (115).

White malleable metallic bead without incrustation;[1] *silver* (117), or *tin* (118).

Red malleable bead without incrustation; *copper*.

White malleable bead and *yellow incrustation; lead* (115).

White brittle bead and *yellow incrustation; bismuth.*

White brittle bead and *white incrustation; antimony.*

Yellow incrustation, becoming *white on cooling; zinc.*

Test for *sulphur* in the fused mass, as in (122).

146. Add the substance by degrees to a bead of borax-glass, and fuse in the outer and inner blowpipe flames (131).

Outer Flame.		Inner Flame.		
Brownish-yellow	glass	**Bottle-green**	glass	*Iron* (135).
Brownish-yellow	"	**Dusky-gray**	"	*Nickel.*
Blue	"	**Colorless**	"	*Copper* (132).
Blue	"	**Blue**	"	*Cobalt.*
Yellowish-green	"	**Emerald-green**	"	*Chromium.*
Purple or pink	"	**Colorless**	"	*Manganese* (137).

147. Cleanse a loop of platinum wire (126) till it no longer tinges the blowpipe flame, moisten it with hydrochloric acid, dip it into the powdered substance, and heat strongly in the inner flame; again moisten with hydrochloric acid, and heat a second time, to be quite sure of the result.

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[1] The white or brownish ash which is always left when charcoal is burnt, must not be mistaken for an incrustation derived from the substance under examination.

Yellow flame	*Sodium.*[1]
Violet flame	*Potassium.*
Orange-red flame	*Calcium.*
Crimson flame	*Strontium.*
Green flame	*Barium.*

148. Test the substance for *silica* and for *manganese* as directed in (137), (138), and (138*a*).

149. Test for *boracic acid* by fusing on platinum wire with fluor spar and bisulphate of potash (139).

CHAPTER VI.

SPECIFIC GRAVITY.

150. By *specific gravity* is meant the relation between the weights of equal bulks of various kinds of matter. It has been found convenient to compare the specific gravities of all solids and liquids with that of water, which is reckoned as 1.000 or 1000. The specific gravity of substances heavier than water is consequently represented by a higher number, and of those which are lighter by a lower number than 1.000; that of lead, for instance, which is more than eleven times heavier than water, is represented by the number 11.44; while that of ether, which is considerably lighter than water, is represented by the number 0.724.[2]

SECTION I.

Specific gravity of solids heavier than water.

151. When the substance is solid and insoluble in water, its specific gravity may be ascertained in the following

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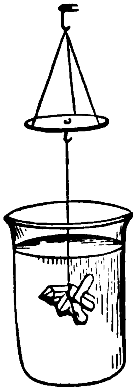
[1] Since very minute quantities of sodium will tinge the flame strongly yellow, it is not safe to conclude that this metal enters to any important extent into the composition of the substance under examination unless there is some other circumstance leading to this belief, such as a resemblance in properties to some known compound of sodium.

[2] See Fowne's Manual of Chemistry, p. 3; or Miller's Chemical Physics, p. 29.

Fig. 51.



manner. Weigh it first in air, taking care to remove any dust or loosely adhering particles. Then suspend it by means of a horse-hair, from a hook attached to the scale-pan, making a small loop at one end of the hair, passing the other end through it, and inclosing the substance in the noose. Thus suspended, it is immersed in water (Fig. 51), and care should be taken that it is covered on all sides by at least half an inch of water. Small bubbles of air frequently adhere to the surface, and these must be brushed off with a feather or camel-hair pencil, as they would tend to buoy it up, and cause the specific gravity to appear too low.

The results may be noted down as follows:—

Weight of the substance in air . . . =
" " in water. . = ________
Loss . . = ========

which number represents the weight of an equal bulk of water. Then by dividing the weight in air by the loss, or the weight of an equal bulk of water, the specific gravity is ascertained.

$$\frac{\text{Weight in air}}{\text{Loss}} = \text{specific gravity.}$$

152. In this way determine the specific gravity of some of the following substances: marble, amber, iron pyrites, sulphate of baryta, jet, lead, zinc, glass, and agate.[1]

SECTION II.

Specific gravity of solids lighter than water.

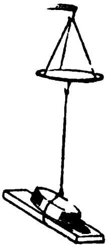
153. If the solid be lighter than water, as cork, a slight modification of the above process is necessary.

---

[1] The following are the specific gravities of these substances, some of which, however, vary considerably. Marble, 2.70; amber, 1.08; iron-pyrites, 4.90; sulphate of baryta, 4.47; jet, 1.30; lead, 11.44: zinc, 7.00; flint glass, 3.30; and agate, 2.60.

Weigh the substance first in air; then select a piece of lead of sufficient size to sink the light body in water when attached to it, and weigh it (the lead) in water, suspending it by means of a hair loop, as before. If now the light substance be inclosed in the same loop with the lead (Fig. 52), and immersed in water, it will be found that they will together weigh less than the lead did alone, owing to the buoyancy of the lighter body, and this difference, when added to the weight of the body in air, is equal to the weight of a corresponding bulk of water.

Fig. 52.



The results may be thus recorded:—

Weight of body in air . . . . = ________
Weight of lead alone in water . . . . . . = ________
Weight of lead with body attached, in water = ________
Difference . . . . . . = ________
Add weight of body in air . . . . = ________
Weight of an equal bulk of water . . . = ________

Having thus obtained the weight of the body in air, and the weight of an equal bulk of water, the specific gravity is calculated as before.[1]

$$\frac{\text{Weight in air}}{\text{Weight of equal bulk of water}} = \text{Specific gravity.}$$

In this way ascertain the specific gravity of wood, cork, and charcoal.[2]

---

[1] The principle of this calculation may not be evident at first sight. Let W represent the weight of the lead alone in water, and w the weight of the lead and cork in water; then w—W would be the weight of the cork alone in water; but the cork being lighter than water, W is greater than w, and hence w—W is a negative quantity, and represents the buoyancy of the cork—*i.e.*, the excess of weight of an equal volume of water over that of the cork. By adding then to this difference the weight of the cork in air, we obtain the weight of an equal bulk of water.

[2] The specific gravity of these substances varies considerably, according to the degree of porosity; the following may be considered as the usual average: wood (beech), 0.85; cork, 0.14; and charcoal 0.2 to 0.5.

SECTION III.

Specific gravity of insoluble powders.

154. When the substance, whose specific gravity we wish to determine, is in the form of powder, or even small lumps, it is clear that some other method must be adopted than those just described. The following is the most simple, and, for common purposes, sufficiently accurate. Counterpoise[1] a small bottle furnished with a stopper; then fill it completely with distilled water, close it with the stopper, taking care that no bubbles of air are left in, and weigh to determine the quantity of water it contains.[2] Having done this, empty the bottle, and dry the inside by warming it and sucking out the air with a tube.

It must now be filled about two-thirds full of the powder to be examined, again weighed, and the bottle then filled cautiously with water, care being taken that all air bubbles are expelled, and that none of the powder is washed out. Again weigh.

From the data thus obtained, the specific gravity may be calculated as follows:—

Weight of the powder and water . . . =
Weight of the powder alone . . . . . =
Difference = weight of water left in the bottle =

Weight of water which fills the bottle . =
Water left in the bottle after the powder was added . . . . =

Weight of water displaced by, and equal in bulk to, the powder . . . . =

Then as before:—

$$\frac{\text{Weight of the powder}}{\text{Weight of water displaced}} = \text{Specific gravity.}$$

In this way ascertain the specific gravity of sand, pounded glass, and shot.[3]

---

[1] This is done by putting shot or strips of lead in a pill-box placed in the opposite scale, until their weight is equal to that of the bottle.

[2] Bottles may be purchased which are made to contain exactly 1000 grains of distilled water.

[3] The specific gravity of sand is about 2.60; flint glass, 3.30; and shot, 11.35.

SECTION IV.

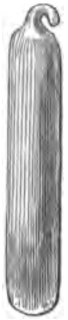
Specific gravity of liquids.

155. With a bottle similar to that used in the last experiment, the specific gravity of liquids may be readily determined. As the space occupied by a given weight of liquid varies with the temperature, or in other words, as the weight of a given volume of any liquid is greater or less as the temperature is lower or higher, it is necessary to observe that the temperature of the liquid during the experiment does not vary much from 62°, which is usually taken as the standard. For the same reason the bottle should not be held in the warm hand during the experiment, as otherwise the heat would cause the liquid to expand, and become specifically lighter.

Counterpoise the bottle, and weigh it full of distilled water; then, by filling it successively with other liquids, weighing, and comparing the different weights with that of water, the volume of liquid being always the same, the specific gravity is obtained by proportion, thus:—

Weight of water : 1.000 : : Weight of liquid : Specific gravity.

Fig. 53.



Specific gravity Glass.

Care must be taken to clean the bottle thoroughly after each experiment, by washing it first with distilled water, and then with a little of the liquid whose density is to be ascertained.

Some of the following may be taken for practice: Alcohol, saturated solution of chloride of sodium, sulphuric acid, nitric acid, hydrochloric acid, solution of ammonia, solution of potash.[1]

156. The specific gravity of liquids may also be determined by another process, which, though not capable of so mnch accuracy as the last, is frequently useful when a specific-gravity bottle is not at hand.

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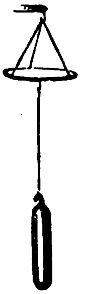
[1] The specific gravity of proof-spirit is 0.920; of oil of vitriol, 1.84; of commercial nitric acid, 1.45; of hydrochloric acid, 1.13 to 1.20; of solution of ammonia, 0.870; of solution of potash, about 1.30.

Take a piece of solid glass rod, about the size of the figure (Fig. 53), with one end drawn out and turned in the blowpipe flame. Weigh it first in the air and then in water, suspending it with a hair-loop (Fig. 54). Then, having wiped it dry between each experiment, weigh it successively in the liquids, the specific gravities of which are to be determined. The difference between the weight of the glass in air and in the liquid, representing in each case the weight of a volume of the liquid equal to that of the glass; and the weight of a similar volume of water being known, the specific gravity may be ascertained by simple calculation.

Fig. 54.



Thus:—

Weight of glass in air . . . .
Weight of glass in liquid . . . .
Loss . . . .

which is the weight of an equal volume of the liquid.

Then by proportion—

$$\left.\begin{matrix}\text{Weight of equal}\\ \text{volume of water.}\end{matrix}\right\} : 1.000 :: \left\{\begin{matrix}\text{Weight of equal}\\ \text{volume of liquid}\end{matrix}\right\} : \left\{\begin{matrix}\text{Specific gravity}\\ \text{of the liquid.}\end{matrix}\right\}$$

Determine in this way the specific gravities of some of the solutions already mentioned, and compare the results with those obtained with the specific gravity bottle.

CHAPTER VII.

HEATING SUBSTANCES IN GASES.

SECTION I.

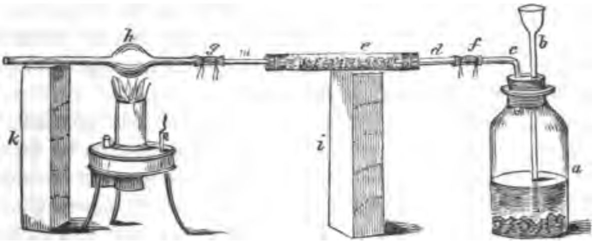
Reduction of metallic oxides by hydrogen.

157. A large number of the metallic oxides are decomposed and reduced to the metallic state when heated in an atmosphere of dry hydrogen gas; and from the facility with which the operation may be performed, and the

accurate results it gives when carefully conducted, it is frequently employed in estimating the quantity of oxygen present in oxidized compounds.

158. The apparatus which is required for the purpose is shown in the figure. (Fig. 55.) The bottle *a* is charged with zinc and dilute sulphuric acid to generate the hydrogen, which is dried while passing over fragments of chloride of calcium in the tube *e*; the gas then passes into the bulb-tube[1] *h*, which contains the oxide to be reduced, the bulb being heated by the lamp placed beneath.

Fig. 55.



Reduction of Oxide of Copper.

159. Take a piece of tubing *e*, about eight or ten inches long, and half an inch internal diameter, and having slightly fused the cut edges in the blowpipe flame, adapt a cork to each end; then with a cork-borer or round file, perforate the corks so as to receive the small tubes *d* and *m*. In using the cork-borers, bore half-way through the centre of the cork, then reverse it, and bore in the opposite direction; the two holes meeting in the centre will be much smoother than if bored directly through.

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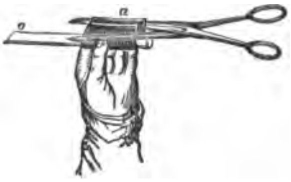
[1] **A piece of plain hard glass tubing (combustion tubing), about half an inch in diameter, provided with a cork and small tube at one end, and drawn out to a point at the other (*d*, Fig. 57), may be substituted for the bulb-tube, provided that care be taken to introduce the powder into its centre, as may be easily done by placing it in a gutter of stiff paper, so as to occupy only about an inch, then sliding the gutter into the tube, and turning this half round upon its axis, so that the powder may fall from the paper on to the glass; the gutter is then carefully withdrawn, without disturbing the powder.**

When the tube is of such a diameter as cannot be exactly matched by any of the cork-borers in the set, the hole should be bored by a smaller one, and afterwards enlarged by means of a round file, until it is of sufficient calibre to admit the tube, which must always fit perfectly tight. Remove one of the corks from the large tube, and push down to the other end a small loose bit of tow or cotton wool, and nearly fill it with fragments of chloride of calcium; put in another bit of tow (the use of which is to prevent any of the smaller fragments falling out), and again fix the cork and small tube.

Next adapt a cork to the bottle, which should have a tolerably wide neck, and bore in it two holes to fit the tubes *b* and *c*, which pass through it, the former reaching nearly to the bottom of the bottle, the latter passing only just through the cork.[1] Put 300 grains of granulated zinc into the bottle, and fix the cork containing the tubes *b* and *c*.

160. In order to connect the different parts of the apparatus together, make two caoutchouc connectors *f* and *g*.[2]

Fig. 56.



Making Caoutchouc Connectors.

This is done by loosely folding a piece of warm sheet caoutchouc about an inch and a half square, round a piece of rod or tubing of the same diameter as the tubes which it is intended to join together, and after having pressed the projecting surfaces together, cutting off with one stroke of a pair of sharp scissors the superfluous ends (Fig. 56); when this is properly done, the cut edges cohere, and when slightly pressed together by the thumb-nails, the junction becomes almost as strong as any other part of the tube. Care must be taken to avoid touching the newly-cut edges, as the least dirt or moisture upon them would prevent them cohering properly together. It is then carefully removed from the rod by moistening the ends in contact with the glass.

---

[1] When a funnel-tube is not at hand, a piece of wide tubing may be passed through the cork, and an ordinary funnel placed in it.

[2] The use of caoutchouc connectors may be dispensed with, if a plain tube be substituted for the bulb-tube; the tube *c* may then be directly inserted into the cork of the chloride of calcium tube, and the tube *m* into that of the plain tube. In some operations, however, the caoutchouc connectors are indispensable, and the student should therefore learn to make them, though the vulcanized tubing is now often substituted for them.

161. Having made two of these connectors, weigh the bulb-tube accurately, and place in the bulb 20 or 30 grains of oxide of copper (CuO); again weigh, to ascertain the weight of oxide operated on, and connect the apparatus as shown in the figure. The caoutchouc tubes should be firmly tied round with strong twine or silk, which should be passed under and over, and tied at each half revolution to insure perfect tightness of the joint. The apparatus being thus arranged, cover the fragments of zinc with about an ounce of water, and add dilute sulphuric acid, by the funnel tube, till a moderate effervescence takes place, and when the gas has been coming over about five minutes, apply a gentle heat to the bulb, and gradually increase it as long as any water is formed.

$$CuO + H = Cu + HO.$$

It is necessary to observe the precaution of not applying the heat immediately, since the apparatus at first contains an explosive mixture of hydrogen and common air, which would, if heat were applied, be in great danger of exploding, and seriously injuring the operator; by allowing five minutes to elapse, however, the whole of the common air is expelled, and the bulb may be heated without danger.

It is advisable to heat the anterior portion of the tube before applying any heat to the oxide of copper, so as to prevent the condensation of any large quantity of moisture, which is apt to crack the tube. A slight inclination towards the open extremity should be given to the bulb-tube, so that any drops of water which may condense shall have no chance of running back upon the hot part of the tube.

162. When the decomposition appears to be complete, no fresh water being produced,[1] expel by heat any moisture that may have condensed in the cool end of the tube, remove the lamp, and allow the bulb-tube to cool; then disconnect the apparatus, and weigh the bulb containing the reduced metallic copper, the loss of weight indicating the quantity of oxygen that has been removed. Ascertain by calculation the proportion of oxygen in 100 parts of the oxide, and compare the experimental result with what is theoretically correct, the atomic weight of copper being 32, that of oxygen 8, and that of the oxide 40.

SECTION II.

Heating substances in an atmosphere of carbonic acid.

163. It is sometimes required in analysis to separate two substances, one of which is volatile at a high temperature, and the other fixed, so that by merely heating the mixture, and weighing before and afterwards, the weight of each ingredient is determined. In some cases, however, it happens that the non-volatile body when heated in atmospheric air, combines with oxygen, forming a volatile compound; so that here it is necessary to conduct the operation in an atmosphere of some gas incapable of combining with it, as hydrogen or carbonic acid. For instance, in the analysis of gunpowder, which consists of a mixture of nitrate of potash (KO,NO_5), sulphur, and charcoal, the nitrate of potash is first dissolved out with water, and the insoluble residue, consisting of sulphur and charcoal, is heated in a current of hydrogen or carbonic acid, when the sulphur, being volatile, is expelled; whereas, if the mixture were to be heated in common atmospheric air, the carbon as well as the sul-

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[1] This is known by holding a piece of cold glass close to the opening at the end of the tube, and observing whether any moisture is condensed upon its surface; if not, it may be inferred that no water is coming off.

phur would disappear, since it would combine with oxygen, and become converted into carbonic acid (CO_2) which is a gas.

164. The apparatus required for this purpose is the same as that used for the reduction of metallic oxides by hydrogen (158). Fill the generating bottle *a* about one-third full of water, and put in some fragments of marble (CaO,CO_2); when the apparatus is arranged, pour in from time to time a little hydrochloric acid through the tube *b*, so as to maintain a moderate effervescence. Weigh the bulb-tube, and put into it about ten grains of the mixture of sulphur and charcoal; weigh a second time, to ascertain how much is used in the experiment, and connect the apparatus together. Allow the gas to come over for about five minutes, in order to displace the common air (which might otherwise cause the loss of some of the charcoal, by conversion into carbonic acid), and then heat the mixture as long as any sulphur is volatilized. As soon as the apparatus is cold, weigh the bulb-tube again, when the loss of weight will represent the quantity of sulphur contained in the mixture. The percentage of sulphur is then ascertained by calculation.[1]

Weight of mixture : loss of weight : : 100 : percentage of sulphur.

SECTION III.

Preparation of perchloride of iron (Fe_2Cl_3).

165. When metallic iron is heated in a current of chlorine gas, the two substances combine, forming perchloride of iron. The chlorine is generated in a retort *a* (Fig. 57), to the beak of which a tube, bent at right angles *b*, should be adapted by means of a perforated cork. The retort is then charged with 700 grains of a mixture of black oxide of manganese (MnO_2) and common salt ($NaCl$), (in the proportion of three parts of the former to four of the latter,) on which should be poured two ounces of water. Remove the tube funnel *b* from the bottle used in the two last experiments (158), and

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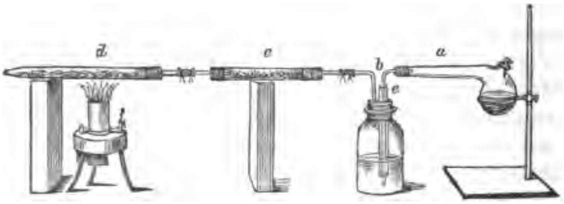
[1] The residue left, when gunpowder is exhausted with water, contains 40 per cent. of sulphur.

substitute a piece of tubing *e*, sufficiently wide to admit the bent tube *b*, and reaching nearly to the bottom of the bottle, which should be filled about a fourth part full of water. The rest of the apparatus is the same as that used in the reduction by hydrogen (158), only substituting the straight tube *d*, which may be six or eight inches long, for the bulb-tube before employed; and put into it thirty grains of clean iron wire.

Fig. 57.



166. When the apparatus is connected together, slowly pour into the retort through a funnel one ounce of strong sulphuric acid (HO,SO_3), to disengage the chlorine from the mixture of manganese and salt; and if the gas does not come over properly apply a very gentle heat.

$$\mathrm{MnO_2+NaCl}+2(HO,SO_3)=MnO,SO_3+NaO,SO_3 \\ +2HO+\mathrm{Cl}.$$

The gas, when generated, passes through the water in the bottle *f*, which retains any hydrochloric acid with which it may be impregnated; and having passed over chloride of calcium in the tube *c*, arrives in the tube containing the iron, in a pure and dry state.

When the apparatus is filled with the chlorine apply a gentle heat to the iron wire, and observe the beautiful scaly crystals of sesquichloride of iron ($\mathrm{Fe_2Cl_3}$), which sublime and condense in the cool end of the tube. Remove a few of the crystals from the tube, and remark with what avidity they absorb moisture from the air when exposed to it for a few minutes.

Dissolve a little of the chloride in distilled water, and add ammonia (NH_3) in slight excess; the brown precipitate which is produced, is hydrated peroxide of iron.

CHAPTER VIII.

VOLUMETRIC ANALYSIS.[1]

ALKALIMETRY, ACIDIMETRY, AND THE VOLUMETRIC DETERMINATION OF IRON.

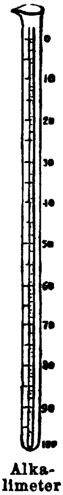
SECTION I.

Alkalimetry.

167. The process of alkalimetry has for its object the determination of the quantity of real alkali or alkaline carbonate in any given sample, and is founded on the principle that the quantity of alkali which is neutralized by a known quantity of acid is always constant and uniform, in obedience to the well-known laws of combination in definite proportions.

For example, forty-nine parts, by weight, of oil of vitriol (HO,SO_3) combine with thirty-one parts of soda (NaO), and when the two substances are brought together in these proportions, the resulting compound (sulphate of soda, NaO,SO_3) is a perfectly neutral salt; but if the relative quantity of acid or alkali be greater or less than those specified, then there will be an *excess* of one of them present, and the solution containing them will be no longer neutral to test paper. Hence it appears that if we have an unknown quantity of pure alkali in a solution, we can, by treating it with an acid of known strength, and observing how much of the acid is required to neutralize it, readily determine the percentage of potash or of soda in any specimen.

Fig. 58.



Alkalimeter Tube.

168. An acid of known strength is prepared by mixing 272 grain measures of concentrated

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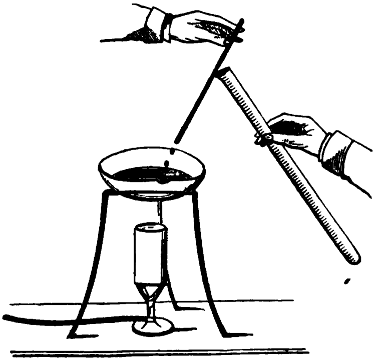
[1] By volumetric analysis is understood the method of determining the quantity of a substance by the use of a measured volume of a reagent of known strength.

sulphuric acid (sp. gr. 1.84) with 20 fluid ounces of distilled water.

In order to ascertain the exact quantity of alkali to which a given volume of this acid corresponds, weigh out 50 grains of pure dry carbonate of soda (which has been strongly heated over a lamp till it suffered no further loss of weight), and dissolve it with the aid of heat in four ounces of water, in a pint evaporating-basin. Add a few drops of a solution of litmus to color the liquid distinctly blue.

Pour into the alkalimeter tube[1] (Fig. 58) 1000 grain

Fig. 59.



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[1] This tube should be capable of holding 1000 grains of distilled water, and should be graduated into 100 equal parts. It is now very generally closed in at the top, and provided with a beak having a small opening from which the liquid may be dropped, and a tube which can be stopped with the finger or thumb, so as to admit, at pleasure, the air required for the efflux of the liquid (burette.) The old alkalimeter tubes were contrived to indicate directly the percentage of alkali or alkaline carbonate in a given sample. A sulphuric acid of specific gravity 1.1268 was employed, and the alkalimeter was marked to denote the exact quantities of this acid required to neutralize 100 grains of potash, soda, and their carbonates respectively. If it were desired to determine the soda in a sample, the alkalimeter was filled with the acid up to the mark *soda*, and then up to zero with water, so that every measure of this diluted acid represented one grain of the alkali, and if 100 grains of the sample were taken, the number of measures required to neutralize it indicated at once the percentage of soda. The method described in the text is of far more general utility.

measures of the dilute sulphuric acid, and allow this to run, drop by drop, down a glass rod held obliquely against the lip[1] of the tube (Fig. 59), into the solution of carbonate of soda, which should be kept hot in order to prevent the liberated carbonic acid from being retained in the liquid. The solution must be stirred at intervals, taking care to redissolve any incrustation on the side of the basin, and the addition of the acid continued until the blue color of the litmus begins to change to red. If on boiling the solution for a minute or two, the blue color returns (from the expulsion of absorbed carbonic acid), another drop of the acid must be added, until the liquid remains permanently red after boiling.[2]

The quantity of acid of the above strength required for this purpose should be about 880 grain measures.

The following proportion will then give the quantity of acid (x) required to neutralize 100 grains of pure soda (NaO):—

$$\underbrace{29.25}_{\substack{\text{NaO in 50 grains}\\ \text{NaO,CO}_2}} : \text{Measures of acid employed} :: 100 : x.$$

and from this, the quantity (y) required for 100 grains of pure potash (KO) may be calculated by the proportion—

$$\underbrace{151.61}_{\text{KO equivalent to 100 NaO}} \left\{\begin{array}{c}\text{Measures of acid required}\\ \text{for 100 NaO}\end{array}\right\} :: 100 : \underset{(KO)}{y}.$$

From these proportions we may deduce the following rule for finding the number of measures of the standard dilute acid which would be required to neutralize 100 grains of pure soda (NaO):—

Multiply the number of measures of acid required to neutralize 50 *grains of carbonate of soda* (NaO,CO_2) *by* 3.419.

---

[1] A little grease rubbed beneath the lip of this tube will prevent any of the acid from trickling down it.

[2] It will be found advantageous to pour a little dilute solution of litmus into another dish, and to redden it with a drop or two of the standard acid (not the measured portion), in order to learn the particular tint of red which the solution is ultimately to acquire.

Again, to find the number of measures of acid required to neutralize 100 grains of pure potash (KO):—

Multiply the number of measures required to neutralize 100 grains of soda (NaO) *by* 0.66.

169. Ascertain the quantity of real alkali (NaO) in a sample of crystallized carbonate of soda (NaO,CO_2+10Aq). For this purpose weigh out 100 grains of the salt, and proceed exactly as above directed in the case of pure carbonate of soda. The number of measures of acid required to neutralize it having been determined, the percentage of soda (x) is calculated by the proportion:—

$$\left.\begin{array}{c}\text{Measures of acid required}\\ \text{by 100 grains NaO}\end{array}\right\} : \overset{\text{NaO}}{100} :: \left.\begin{array}{c}\text{Measures of acid used}\\ \text{in the experiment.}\end{array}\right\} : x.$$

The atomic weight of crystallized carbonate of soda being 143, and that of soda 31, a hundred grains of the pure crystals should contain 21.67 of soda.

170. Determine the percentage of potash (KO) in the crystallized bicarbonate of potash ($KO,HO,2CO_2$), and compare the result with that required by theory, which may be deduced from the following data:—

$$K=39,\ O=8,\ H=1,\ C=6.$$

SECTION II.

Acidimetry.

171. The process of alkalimetry being well understood, that of acidimetry will require but little explanation, as its principle is precisely analogous to that which has been described (167). In the former process the object was to determine the quantity of alkali by the quantity of acid which it was capable of neutralizing; in acidimetry, we have to ascertain the amount of real acid in any solution containing it in an uncombined form. When the acid under examination forms with lime, a salt that is soluble in water, its strength may be ascertained by determining the quantity of marble or carbonate of lime (CaO,CO_2) which a given weight of it decomposes and dissolves. This process will serve for nitric, hydrochloric and acetic acids.

172. Determine the percentage of nitric acid (HO,NO_5) in a specimen of the liquid acid.

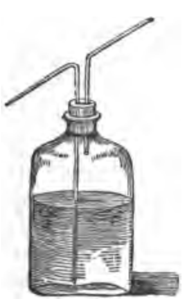
Weigh out 150 grains of pounded marble, put it into an evaporating basin, and cover it with about two ounces of distilled water. Pour a little of the acid into a glass, and thence, by means of a dropping tube (97), transfer exactly 100 grains of it into a previously counterpoised capsule. Add this in successive portions to the marble, avoiding too large an addition at once, lest the effervescence should be so violent as to cause some of the liquid to be projected over the sides of the basin and lost.

When the whole of the acid has been added, wash out the dish which contained it two or three times with distilled water, and add the washings to the marble; stir the mixture repeatedly with a glass rod, and when the effervescence appears to have nearly ceased, heat it gently over a lamp until the solution is no longer acid.

$$\mathrm{CaO,CO_2}+HO,NO_5=CaO,NO_5+\mathrm{CO_2}+HO.$$

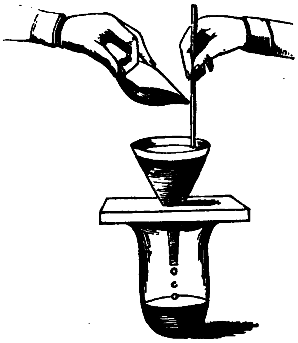
173. While this is going on, fit up a washing bottle (Fig. 60), the tubes for which have already been prepared

Fig. 60.



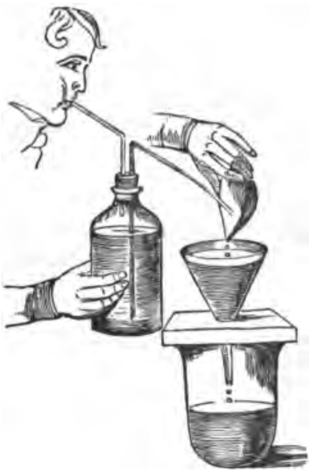
Washing Bottle.

Fig. 61.



(94). Two holes must be bored in the cork to fit the tubes, which must be fixed in the manner shown in the figure. Then prepare a filter, according to the directions already given (66); and having moistened it with distilled water, support it over a beaker glass by means of a retort stand or perforated block of wood (Fig. 61). Pour the solution from the evaporating basin down a glass rod into the filter, directing the stream so that it may fall upon the sloping side and not into the apex, lest its force should injure or break through the paper; wash the last portions of marble out of the basin by means of the washing bottle, holding the basin in a nearly vertical position (Fig. 62). When the liquid has for the most part passed through the filter, wash the latter with water from the washing bottle, directing the stream just below the upper edge of the filter, and continuing to wash until a drop of the filtered liquid, when evaporated on a strip of glass or platinum foil leaves no trace of solid matter. When this is the case, we may be sure that the whole of the soluble nitrate of lime (CaO,NO_5) has been washed out, and that nothing remains but the portion of marble which was not decomposed. The filter containing the residue is now very carefully raised from the funnel, the operator lifting the filter by its double side, and laying it down on the single side in a large shallow evaporating dish; the filter is tenderly opened, without tearing it, and the dish placed over a very small gas flame, so as to dry the filter without scorching it; when it is perfectly dry, empty every particle of the marble into a porcelain capsule (previously counterpoised), and weigh it. Its weight is deducted from that of the whole of the marble employed; the difference being of course the weight of that which has been dissolved by the acid. Now since every equivalent of carbonate of lime which has been dissolved, indicates the

Fig. 62.



presence of an equivalent of nitric acid, the quantity of nitric acid in the 100 grains employed may be ascertained by the following proportion:—

$$\overbrace{50}^{\substack{\text{Atomic weight of}\\ \text{carbonate of lime,}}} : \overbrace{63}^{\substack{\text{Atomic weight of}\\ \text{nitric acid.}}} :: \left\{\begin{array}{c}\text{Quantity of mar-}\\ \text{ble dissolved,}\end{array}\right\} : x = \left\{\begin{array}{l}\text{Percentage}\\ \text{of nt. acid.}\end{array}\right.$$

174. Ascertain the percentage of hydrochloric acid (HCl) in a specimen of the liquid acid. Proceed exactly as in the previous experiment with nitric acid (173), only in the calculation substitute the atomic weight of hydrochloric acid 36.5, for that of nitric, thus:—

$$50 : 36.5 :: \text{Quantity of marble dissolved} : x.$$

175. In the determination of the strength of acids which do not form, with lime, salts easily dissolved by water, the method just described will not, of course, give accurate results, and the method described in (168) is then adopted, the quantity of real acid being deduced from the amount required to neutralize a given weight of carbonate of soda.

176. Determine the percentage of sulphuric acid (HO,SO_3) in a specimen of the dilute acid, by the method described in (168), using 53 grains of pure carbonate of soda. The number of measures of the dilute acid required for neutralization will be known to contain 49 grains of sulphuric acid (HO,SO_3), since 53 is the equivalent number of carbonate of soda, and 49 that of the acid. It only remains, therefore, to calculate the percentage:—

$$\left.\begin{array}{c}\text{Measures of dilute acid}\\ \text{employed}\end{array}\right\} : 49 :: 100 : \left\{\begin{array}{l}\text{Percentage of}\\ \text{sulphuric acid}\end{array}\right.$$

177. Determine the strength of nitric ($HO,NO_3=63$) and hydrochloric ($HCl=36.5$) acids by this method, and compare the results with those obtained by the decomposition of marble.

SECTION III.

Volumetric Determination of Iron.

178. The determination of the quantity of iron in its ores being frequently required, various methods have been devised for effecting it with the greatest rapidity and accuracy. One of the easiest of these is here described as affording an excellent example of volumetric analysis. It depends upon the property possessed by the proto-salts of iron, of decolorizing the purple-red solution of permanganate of potash (KO,Mn_2O_7), by reducing the permanganic acid to protoxide of manganese (MnO), the solution of which is colorless.

$$Mn_2O_7 + 10FeO = 2MnO + 5Fe_2O_3$$

179. A standard solution of permanganate of potash is first prepared by dissolving 60 grains of the pure crystallized salt in 20 measured ounces of distilled water.

To determine the exact amount of iron to which a given volume of this solution corresponds, dissolve 10 grains of iron wire placed in a small flask,[1] in one measured ounce of dilute sulphuric acid (containing one-fifth of its bulk of the strong acid); the operation may be hastened by a gentle heat, and as soon as the whole of the iron is dissolved, the solution is carefully poured into an evaporating basin and the flask rinsed out with water, the rinsings being added to the solution, which is then diluted with ten ounces of water. One thousand grains of the solution of permanganate of potash are measured out in a graduated tube or burette (168), and very carefully added, by degrees, to the solution of iron, with occasional stirring, until a permanent rose tint becomes perceptible in the solution, showing that a slight excess of the permanganate has been added. At least 830 grains of the solution would be required for this purpose. The number of grain measures employed having been carefully noted, the quantity of iron (x) represented by 1000 grains of the solution is calculated and recorded:—

$$\text{Number of measures employed} : \overset{Fe}{10} :: 1000 : x.$$

---

[1] A Florence flask will answer the purpose.

180. Determine the percentage of iron in a specimen of the peroxide (Fe_2O_3).

Dissolve 15 grains of the peroxide in a flask, with the aid of heat, in three drachms of hydrochloric acid diluted with 1 drachm of water.

The solution of perchloride of iron (Fe_2Cl_3) thus obtained must now be reduced to the state of protochloride ($FeCl$), which may be effected by the addition of sulphite of soda (NaO,SO_2).

$$Fe_2Cl_3+NaO,SO_2+HO=2FeCl+NaO,SO_3+HCl.$$

Dilute the solution in the flask with two ounces of water, and add solution of carbonate of soda by degrees, with frequent agitation, until the effervescence is no longer very brisk, the color of the liquid has become darker, and the precipitate formed at first does not instantly redissolve. (For this purpose it will generally be found necessary to add 1 fluid ounce of solution of carbonate of soda, containing 70 grains of the dry carbonate, or 189 grains of the crystallized carbonate.) Boil the solution, and if it should become turbid, add hydrochloric acid, very carefully, drop by drop, until it becomes clear again. (This should never require more than 30 drops of the acid.)

To the boiling solution, add solution of sulphite of soda, drop by drop, until the color has either entirely disappeared or has changed to a pale green (14 grains of the crystallized sulphite of soda (NaO,SO_2+7Aq) dissolved in 2 drachms of water will generally be found to suffice). To the decolorized liquid add 1 drachm of hydrochloric acid, and continue the ebullition until the odor of sulphurous acid is no longer perceptible at the mouth of the flask, which will be the case in ten or fifteen minutes.

Remove the flame from under the flask, and pour in four ounces of water; pour the liquid into a pint evaporating dish, and rinse the flask out with 6 ounces of water, pouring the rinsings into the dish.

The solution is now tested with the permanganate of potash, as directed in (179).

From the number of measures of the permanganate employed, the amount of iron (x) present is calculated by the proportion:—

$$1000 : \left\{ \begin{matrix} \text{Quantity of iron represented by} \\ \text{1000 grains of permanganate} \end{matrix} \right\} :: \left\{ \begin{matrix} \text{Measures of the} \\ \text{permanganate used} \end{matrix} \right\} : x.$$

PART II.

RECOGNITION OF METALS AND NON-METALLIC BODIES IN THEIR MOST FREQUENT FORMS OF COMBINATION.

CHAPTER I.

SECTION I.

Introductory.

181. QUALITATIVE analysis has for its object the determination of the elements or compounds which are contained in any given substance; and those elements and compounds are recognized by certain characteristic appearances which they present when exposed to the action of tests or reagents, or when otherwise treated, as when submitted to heat, &c.

Before proceeding, therefore, to the more complicated processes of analysis, it is advisable that the student should make himself familiar with the action of reagents on the compounds most commonly met with in such investigations, in order to enable him properly to interpret the language in which Nature, through his experiments, replies to his inquiries.

With this purpose in view, he should not merely apply his tests, and superficially note whether a precipitate is or is not formed, but he should endeavor to impress on his recollection the exact appearance which it presents, both as to color and also as to physical structure; whether it is crystalline, curdy, or gelatinous; whether it separates immediately from the solution, or requires time for its development; as well as the action of solvents (as acids and alkalies) upon it. Besides the increased facility which he will thus gain in making subsequent experiments, he will be acquiring habits of close and accurate observation, which will be of infinite value to him, not only in pursuing the study of chemistry, but in almost every occupation of life.

SECTION II.

Analytical Classification.

182. In describing the action of reagents, and the rudiments of chemical analysis, all the rarer substances will be omitted, as they would only tend to confuse the student.[1] The following are those which will be treated of, as being most commonly met with in analysis. The metals are classified according to their behavior with *hydrosulphuric acid* (HS), *hydrosulphate of ammonia* (NH_4S,HS), and *carbonate of soda* (NaO,CO_2). Those in Class IV. are precipitated as sulphides from acidified solutions by hydrosulphuric acid; those in Class III. are not affected by hydrosulphuric acid when an excess of hydrochloric acid is present, but are thrown down either as sulphides or oxides when their neutral solutions are treated with hydrosulphate of ammonia; those in Class II. are not precipitated by either hydrosulphuric acid or hydrosulphate of ammonia, but are thrown down as carbonates, by carbonate of ammonia or of soda: and those in Class I. are unaffected by any of those reagents.

METALS AND THEIR PRINCIPAL OXIDES.

Class I.

Metals.	Oxides.
Potassium (K) . .	Potash (KO)
Sodium (Na) . . .	Soda (NaO)
Ammonium (NH_4) . .	Oxide of Ammonium (NH_4O)

---

[1] In the Appendix will be found a table showing the behavior of most of the rarer substances with reagents.

Class II.

Metals.	Oxides.
Barium (Ba)	Baryta (BaO)
Strontium (Sr)	Strontia (SrO)
Calcium (Ca)	Lime (CaO)
Magnesium (Mg)	Magnesia (MgO)

Class III.

Aluminum (Al)	Alumina (Al_2O_3)
Chromium (Cr)	Oxide of Chromium (Cr_2O_3)
	Chromic Acid (CrO_3)
Zinc (Zn)	Oxide of Zinc (ZnO)
Manganese (Mn)	Protoxide of Manganese (MnO)
Iron (Fe)	Protoxide of Iron (FeO)
	Peroxide of Iron (Fe_2O_3)
Cobalt (Co)	Oxide of Cobalt (CoO)
Nickel (Ni)	Oxide of Nickel (NiO)

Class IV.

Arsenic (As)	Arsenious Acid (AsO_3)
	Arsenic Acid (AsO_5)
Antimony (Sb)	Oxide of Antimony (SbO_3)
Tin (Sn)	Protoxide of Tin (SnO)
	Binoxide of Tin (SnO_2)
Silver (Ag)	Oxide of Silver (AgO)
Lead (Pb)	Oxide of Lead (PbO)
Mercury (Hg)	Suboxide of Mercury (Hg_2O)
	Oxide of Mercury (HgO)
Copper (Cu)	Oxide of Copper (CuO)
Bismuth (Bi)	Oxide of Bismuth (BiO_3)

NON-METALLIC BODIES AND THEIR PRINCIPAL ACIDS.

Non-metallic bodies.	Acids.
Nitrogen (N)	Nitric Acid (NO_5)[1]
Sulphur (S)	Hydrosulphuric Acid (HS)
	Sulphurous Acid (SO_2)
	Hyposulphurous Acid (S_2O_2)
	Sulphuric Acid (SO_3)
Phosphorus (P)	Phosphoric Acid (PO_5)
Carbon (C)	Carbonic Acid (CO_2)
	Oxalic Acid (C_2O_3)
	Hydrocyanic Acid (HC_2N)
	Hydroferrocyanic Acid ($H_2[C_6N_3]Fe$)
	Tartaric Acid ($C_8H_4O_{10}$)
	Citric Acid ($C_{12}H_5O_{11}$)

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[1] The composition of the acids containing oxygen is here given as they occur in their salts, omitting the water of hydration usually associated with them in their free state.

Non-metallic Bodies.	Acids.
	Gallic Acid ($C_{14}H_3O_7$)
	Tannic Acid ($C_{54}H_{22}O_{34}$)
	Benzoic Acid ($C_{14}H_5O_3$)
	Acetic Acid ($C_4H_3O_3$)
Boron (B)	Boracic Acid (BO_3)
Silicon (Si)	Silicic Acid (SiO_2)
Chlorine (Cl)	Hydrochloric Acid (HCl)
	Hypochlorous Acid (ClO)
	Chloric Acid (ClO_5)
Iodine (I)	Hydriodic Acid (HI)
Fluorine (F)	Hydrofluoric Acid (HF)

183. Should the student find that the action of any test does not agree with that described, it may be owing to some impurity contained in the test liquid, in which case he may examine it in the manner described in the section on reagents.

It will be observed that, in general, only those reactions are set down which admit of practical application in the systematic course of analysis, those which possess merely a general interest being omitted.

The student is cautioned against supposing that any reaction which is ascribed to one substance, and not to others of the same group, may serve as a characteristic test for that substance, since such an application will always receive especial notice, and the reaction is omitted in the case of the other substances, merely because it is not applied in the systematic course of analysis.

CHAPTER II.

METALS BELONGING TO CLASS I.

Potassium, Sodium, and Ammonium.

184. The three metals belonging to this class are chiefly characterized by the solubility in water of most of their compounds, and the consequent difficulty of obtaining them in an insoluble form, and of separating them from one another in the shape of precipitates. They are distinguished from all other metals by producing no precipi-

tate when tested with either of the three classifying tests —viz: hydrosulphuric acid, hydrosulphate of ammonia, and carbonate of ammonia, their sulphides and carbonates being all soluble in water.

Solutions of the oxides (alkalies) of these metals, and of their carbonates, are alkaline to test-paper, turning reddened litmus blue, and turmeric brown.

SECTION I.

Potassium (K).

185. Evaporate a drop of the solution of a compound of potassium (chloride of potassium or nitrate of potash, for example) upon a slip of thin glass or of platinum foil, and observe that the dry residue is *fixed*—that is, will not volatilize upon the continued application of heat.

186. (C)[1] Place a drop of the solution (*e. g.*, chloride of potassium), with a glass rod, upon a piece of glass, add a drop of *hydrochloric acid*, and another of *bichloride of platinum* ($PtCl_2$). Stir them well together with the rod, and observe the formation of a yellow crystalline precipitate of the double chloride of platinum and potassium ($KCl,PtCl_2$), especially upon the lines where the glass has been rubbed with the rod.

If the solution of potassium be very dilute, a considerable time may be required for the production of this precipitate.

187. Repeat this experiment with iodide of potassium (KI); notice the red color due to biniodide of platinum (PtI_2), and the delay in the appearance of the precipitate.

188. (C) Test a drop of the solution of chloride of potassium, upon a piece of glass, with a strong solution of *tartaric acid* ($2HO,C_8H_4O_{10}$). On stirring briskly, a very crystalline white precipitate of bitartrate of potash ($KO,HO,C_8H_4O_{10}$) will be formed. A drop of hydrochloric acid will redissolve it, so that this test can only be depended on when the solution is neutral or alkaline. Be-

[1] Tests marked (*C*) (characteristic) are those generally employed in analysis, and should therefore receive particular attention.

fore testing an acid solution with tartaric acid, it should be neutralized with carbonate of soda.

[The blowpipe test will be found at (127).]

SECTION II.

Sodium (Na).

189. Evaporate a drop of a solution containing sodium (*e. g.*, sulphate of soda) upon glass or platinum; the dry residue will not volatilize when heated.

190. (C) Test a drop of the solution on a piece of glass with *antimoniate of potash* (KO,SbO_5).[1] On stirring briskly, a white crystalline precipitate of antimoniate of soda (NaO,SbO_5) will be deposited.

Test a drop of any dilute acid (as hydrochloric) with antimoniate of potash, and observe that it gives an amorphous precipitate of hydrated antimonic acid (HO,SbO_5), showing that an acid solution must not be tested for soda until it has been neutralized with carbonate of potash.

[The blowpipe test for sodium will be found at (126).]

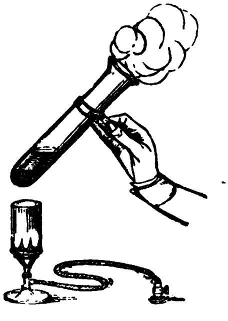
SECTION III.

Ammonium[2] (NH_4).

191. (C) Evaporate a drop of solution of chloride of ammonium upon a piece of glass or of platinum foil, and continue to heat the dry residue; it will entirely volatilize.

192. (C) Boil some solution of chloride of ammonium in a test-tube;[3] observe that no smell of ammonia is perceived, but add a little *potash*, and the liberated ammonia will at once be recognized

Fig. 63.



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[1] The bimetantimoniate of potash of Frémy, who represents it by the formula KO,HO,SbO_5.

[2] It will be remembered that this metal is not known in the uncombined state.

[3] When a liquid is to be boiled in a test-tube, the latter may be held in a loop of paper or cloth, as shown in Fig. 63.

by its odor and by its restoring the blue color to moist red litmus paper, which should only be exposed to the vapor, and not allowed to touch the tube.

$$NH_4Cl+KO=KCl+NH_3+HO.$$

193. Test a drop of solution of chloride of ammonium upon a slip of glass with *bichloride of platinum*, which will produce a precipitate of the double chloride of platinum and ammonium ($NH_4Cl,PtCl_2$) very similar to that obtained with a compound of potassium (186).

194. Mix a little solution of carbonate of ammonia[1] with *tartaric acid* upon a piece of glass; observe how easily the precipitate of bitartrate of ammonia ($NH_4O,HO,C_8H_4O_{10}$) might be mistaken for bitartrate of potash (188).

Summary of Class I.

195. The three metals of the alkalies may be distinguished from other metals by their producing no precipitate with either hydrosulphuric acid, hydrosulphate of ammonia, or an alkaline carbonate; one or other of which causes, as we shall presently see, a precipitate with all the other metals. Hence, if we have a solution which we know to contain a metal,[2] and we find no precipitate produced in it on the application of those tests, we conclude that the metal present is either potassium, sodium, or ammonium.

For the purpose of distinguishing between the three metals themselves, we may first test for ammonium, by heating with potash (192). If this is absent, add to a tolerably concentrated solution on a slip of glass, some bichloride of platinum or tartaric acid (186, 188), which will enable us to distinguish between potassium and sodium. If these tests give no precipitate, it is probable that the metal is sodium; which may be confirmed by the behavior of the solution with antimoniate of potash (190), and by the color which the salt imparts to the blowpipe flame (126).

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[1] If chloride of ammonium were employed it would be difficult to obtain a precipitate, since it is very readily soluble in the hydrochloric acid liberated by the decomposition.

[2] This may be inferred when a solution which is neutral or alkaline to test-paper leaves a *fixed* residue on evaporation. Of course, should the residue volatilize, it does not prove the absence of *all* metals.

CHAPTER III.

METALS BELONGING TO CLASS II.

Barium, Strontium, Calcium, and Magnesium.

196. These metals are distinguished from the preceding by the insolubility of many of their salts, especially their carbonates and phosphates; so that when treated with carbonate or phosphate of soda, they furnish copious precipitates.

SECTION I.

Barium (Ba).

197. Pour a few drops of chloride of barium ($BaCl$) into a test-tube, and add a little *carbonate of soda*, which will give a white precipitate of carbonate of baryta (BaO,CO_2).

$$BaCl + NaO,CO_2 = NaCl + \mathrm{BaO,CO_2}.$$

The precipitate will be easily dissolved on adding dilute hydrochloric acid. Carbonate of ammonia produces the same precipitate, but since the sesquicarbonate ($2NH_4O,3CO_2$) is generally used, it is necessary to boil the solution in order to expel the extra equivalent of carbonic acid, which would retain some of the carbonate of baryta in solution.

198. Dilute *sulphuric acid* (HO,SO_3) produces in solutions containing barium, even though they be very dilute, a copious white precipitate of sulphate of baryta (BaO,SO_3).

$$BaCl + HO,SO_3 = \mathrm{BaO,SO_3} + HCl.$$

This precipitate is insoluble in acids.

199. (C) Solution of *sulphate of lime* (CaO,SO_3) causes an immediate precipitate of sulphate of baryta. This is the most convenient mode of applying a very dilute solution of a sulphate, since sulphate of lime requires about 500 times its weight of water to dissolve it.

200. (C) *Hydrofluosilicic acid* (HF,SiF_2) causes a white highly crystalline precipitate of silicofluoride of barium (BaF,SiF_2), insoluble in dilute acids.

[For the blowpipe test for barium, see (130).]

SECTION II.

Strontium (Sr).

A solution of nitrate of strontia (SrO,NO_5) may be used.

201. *Alkaline carbonates* act as with solutions of barium (197), carbonate of strontia (SrO,CO_2) being produced.

202. *Sulphuric acid* (HO,SO_3) produces a white precipitate of sulphate of strontia (SrO,SO_3) immediately, if the solution is not very dilute, and after standing a short time if it is so.

$$SrO,NO_5 + HO,SO_3 = \mathrm{SrO,SO_3} + HO,NO_5.$$

203. (C) Solution of *sulphate of lime* (CaO,SO_3) gives no immediate precipitate in solutions of strontium, but if allowed to stand, sulphate of strontia gradually separates. Heat hastens the precipitation. Strontium may thus be distinguished from barium (199).

[For the blowpipe test, see (129).]

SECTION III.

Calcium (Ca).

A solution of chloride of calcium ($CaCl$) may be used.

204. *Carbonate of ammonia* (NH_4O,CO_2) produces a white precipitate of carbonate of lime (CaO,CO_2) which becomes far less voluminous on heating the solution; it dissolves very readily in hydrochloric acid.

205. *Sulphuric acid* (HO,SO_3), when added to concentrated solutions containing calcium, gives an immediate white precipitate of sulphate of lime (CaO,SO_3+2Aq).

$$CaCl+HO,SO_3=CaO,SO_3+HCl.$$

If the solution is not concentrated, the precipitate may not appear at once, but will gradually separate in the form of minute crystals; and if the solution is very dilute, no precipitation will take place, because the sulphate of lime, being soluble in about 500 times its weight of water, remains dissolved if sufficient water is present. In solutions of sulphate of lime, of course no precipitate is produced by either of these reagents.

206. (C) After having thrown down the sulphate of lime, pour the mixture on a filter, and test the filtered solution with *ammonia and oxalate of ammonia* (207); sufficient of the sulphate will have been retained in solution to give a very perceptible precipitate with the oxalate.

207. (C) *Oxalate of ammonia* (NH_4O,C_2O_3) is an extremely delicate test for lime, though it does not serve to distinguish it from baryta and strontia, which are also precipitated by it. When added to a solution containing it even in a highly diluted state, a copious white precipitate of oxalate of lime (CaO,C_2O_3+2Aq) is produced.

$$CaCl+NH_4O,C_2O_3=CaO,C_2O_3+NH_4Cl.$$

It is necessary that the solution should contain no excess of acid, as the oxalate of lime is soluble in acid solutions; acetic acid, however, does not dissolve it.

[For the blowpipe test, see (128).]

SECTION IV.

Magnesium (Mg).

A solution of the sulphate (MgO,SO_3) is the most convenient for the following experiments.

208. Ammonia (NH_3), when added to a neutral solution of magnesium, separates a portion of it in the form

of hydrate of magnesia (MgO,HO), which appears as a bulky white precipitate.

$$MgO,SO_3+NH_3+2HO=MgO,HO+NH_4O,SO_3.$$

The rest of the magnesium remains in solution as a soluble double salt of ammonia and magnesia ($NH_4O, MgO,2SO_3$).

209. When *chloride of ammonium* (NH_4Cl) has been added to a solution containing magnesium, *ammonia* no longer produces any precipitate, since the hydrate of magnesia is soluble in chloride of ammonium.

210. *Carbonate of soda* (NaO,CO_2) gives, especially on boiling, a white precipitate, consisting of basic carbonate of magnesia ($4MgO,3CO_2+4Aq$). A portion of the magnesia remains in solution as bicarbonate, which when boiled is decomposed, and the neutral carbonate (MgO, CO_2) being insoluble is precipitated. Ammoniacal salts, if present, prevent the formation of these precipitates, and redissolve them if subsequently added.

211. *Carbonate of ammonia* (NH_4O,CO_2) gives no precipitate unless the solution is boiled. The previous addition of chloride of ammonium entirely prevents the precipitation.

212. (C) *Phosphate of soda* ($2NaO,HO,PO_5$) gives a white precipitate of phosphate of magnesia ($2MgO,HO, PO_5$) provided the solution is not very dilute, and especially on boiling.

The addition of *chloride of ammonium* and *ammonia* to the magnesian solution, renders the phosphate of soda a far more delicate test than when used alone, because under those circumstances the double phosphate of ammonia and magnesia ($2MgO,NH_4O,PO_5+12Aq$) is produced, which is less soluble than the phosphate of magnesia, and is consequently thrown down from a more dilute solution than would furnish a precipitate with phosphate of soda alone. If the solution is very dilute, the precipitate does not appear at once, but if allowed to stand some little time, a crystalline deposit of the double phosphate gradually separates. Agitation of the liquid with a glass rod hastens the formation of this precipitate; and it is remarkable that if the tube be rubbed at all with the rod during agitation, lines of minute crystals are there first deposited. The same phenomenon occurs in the case of the bitartrate of potash and others, in which the precipitate is slowly deposited from a dilute solution.

As the double phosphate is readily soluble in an excess of acid, and slightly so in water, it is necessary that the solution should be pretty strongly ammoniacal.

It will be observed that in this test, the effect of ammoniacal salts in the solution is the reverse of that before described-(209). When mixed with ammoniacal salts, indeed, magnesia can be precipitated only by a soluble phosphate.

Salts of barium, strontium, and calcium being also precipitated by phosphate of soda, this test cannot be relied on unless the absence of those metals has been ascertained.

213. *Baryta water* (*BaO* in water) throws down a white precipitate of hydrate of magnesia (MgO,HO). If the sulphate of magnesia be used, the insoluble sulphate of baryta (BaO,SO_3) will be thrown down at the same time.

$$MgO,SO_3+BaO,HO=MgO,HO+BaO,SO_3.$$

Summary of Class II.

214. Supposing that we had in solution a salt of one of the metals belonging to this class—viz: barium, strontium, calcium, or magnesium, we should be able, without any difficulty, by applying a few of the most characteristic tests, to ascertain which individual of the class it was. Thus we should find that a solution of hydrosulphate of ammonia gave no precipitate, and that an alkaline carbonate gave a white one either at once or on boiling; from which we should infer that the metal belongs to Class II. We might then test it with a solution of sulphate of lime, which would tell us whether barium or strontium was present (203); if not, we should add to a dilute solution a little oxalate of ammonia, which, if the metal were calcium, would throw it down as oxalate (207). If neither of these tests gave any indication, chloride of ammonium, ammonia, and phosphate of soda

should be added, when, if the base were magnesia, the double phosphate of ammonia and magnesia would be precipitated (212).

Before finally deciding, however, that the base is either of these, it is always necessary to apply other confirmatory tests in addition to those just mentioned.

CHAPTER IV.

METALS BELONGING TO CLASS III.

Aluminum, Chromium, Zinc, Manganese, Iron, Nickel, and Cobalt.

The metals of the third class are distinguished from those of the first and second, in being precipitated from their neutral solutions by hydrosulphate of ammonia; and from those of the fourth class in not being precipitated when their solutions, containing a slight excess of acid, are treated with hydrosulphuric acid.

SECTION I.

Aluminum (Al).

A solution of sulphate of alumina ($Al_2O_3,3SO_3$) may be used.

215. *Hydrosulphate of Ammonia* (NH_4S,HS) gives a very transparent, gelatinous precipitate of hydrate of alumina ($Al_2O_3,3HO$), and hydrosulphuric acid is at the same time liberated.

$$Al_2O_3,3SO_3+3(NH_4S,HS)+6HO=Al_2O_3,3HO+3(NH_4O,SO_3)+6HS.$$

216. *Ammonia* (NH_3) throws down a similar gelatinous precipitate, consisting chiefly of hydrate of alumina ($Al_2O_3,3HO$) with a small admixture of a basic salt of

alumina; which is almost insoluble in an excess of ammonia.

$$Al_2O_3,3SO_3+3NH_3+6HO=\mathrm{Al_2O_3,3HO}+3(NH_4O,SO_3).$$

217. (C) *Potash* (KO) also gives a precipitate of hydrate of alumina, which, like that caused by ammonia, usually contains a little basic salt; it differs from it, however, in being entirely soluble in an excess of the precipitant. If the solution in potash be mixed with chloride of ammonium (NH_4Cl), the alumina is again precipitated.

$$Al_2O_3,3SO_3+3(KO,HO)=\mathrm{Al_2O_3,3HO},+3(KO,SO_3).$$

218. *Phosphate of Soda* ($2NaO,HO,PO_5$) produces a gelatinous precipitate of phosphate of alumina (Al_2O_3,PO_5); soluble in hydrochloric acid, and reprecipitated by ammonia; readily soluble in potash, and reprecipitated by acetic acid.

SECTION II.

Chromium (Cr).

A. *As Oxide of Chromium* (Cr_2O_3) *or some corresponding compound.*

A solution of sulphate of chromium ($Cr_2O_3,3SO_3$) may be used.

219. *Hydrosulphate of ammonia* (NH_4S,HS) throws down a greenish-blue precipitate of hydrated oxide of chromium ($Cr_2O_3,10HO$).

220. (C) *Ammonia* (NH_3) also produces the same precipitate ($Cr_2O_3,10HO$), a small portion of which redissolves in an excess of ammonia, forming a pale pinkish solution, but is again precipitated when the mixture is boiled.

221. (C) *Potash* (KO) also throws down the hydrated oxide, which is soluble in excess, forming a green solution; if this solution be boiled for a minute or two, the hydrated oxide is again precipitated.

222. (C) Oxide of chromium, when fused (on platinum

foil) with nitrate of potash and a little carbonate of soda (the blowpipe flame being directed upon the under surface of the foil), yields a yellow mass. Here a portion of the oxygen of the nitric acid combines with the oxide of chromium (Cr_2O_3), converting it into chromic acid (Cr_2O_6), or rather (CrO_3), which combines with the potash or soda, forming an alkaline chromate (KO,CrO_3). If the mass be dissolved in water and acidulated with a little acetic acid, the solution will give with acetate of lead a bright yellow precipitate of chromate of lead (PbO, CrO_3).[1]

[For another blowpipe test, see (131).]

B. *As Chromic Acid* (CrO_3).

A solution of bichromate of potash ($KO,2CrO_3$) may be used.

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223. *Hydrochloric* and *hydrosulphuric acid* change the red color of the solution to green (sesquichloride of chromium Cr_2Cl_3), the solution becoming turbid from separation of sulphur.

$$2CrO_3+3HCl+3HS=Cr_2Cl_3+6HO+S_3.$$

224. *Hydrosulphate of ammonia* (NO_4S,HS) gives a greenish-gray precipitate, consisting of a mixture of sesquioxide of chromium and sulphur.

225. (C) *Chloride of Barium* ($BaCl$) throws down a yellow precipitate of chromate of baryta (BaO,CrO_3), soluble in dilute hydrochloric acid.

226. (C) *Nitrate of silver* (AgO,NO_5) produces a purple-red precipitate of chromate of silver (AgO,CrO_3), soluble, though with some difficulty, in nitric acid.

227. (C) *Acetate of lead* ($PbO,C_4H_3O_3$) gives a yellow precipitate of chrome yellow (PbO,CrO_3) insoluble in acetic acid.

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[1] It is advisable to boil the acetic solution before testing with acetate of lead, in order to decompose any nitrate of potash.

SECTION III.

Zinc (Zn).

A solution of sulphate of zinc (ZnO,SO_3) may be used.

228. *Hydrosulphuric acid* (HS), when added to a solution of a neutral salt of zinc, causes the precipitation of a portion of it as sulphide (ZnS); but if the solution be mixed with hydrochloric acid (HCl), hydrosulphuric acid will produce no precipitate.

229. (C) *Hydrosulphate of ammonia* (NH_4S,HS) gives a precipitate of sulphide (ZnS), which, if the zinc salt be pure, is white;[1] but if, as is frequently the case, any iron is present, the precipitate will be more or less colored, owing to the admixture of a little of the black sulphide of iron (FeS).

$$ZnO,SO_3 + NH_4S,HS = \mathrm{ZnS} + NH_4O,SO_3 + \mathrm{HS}.$$

230. (C) *Ammonia* (NH_3) throws down a white gelatinous precipitate of hydrated oxide of zinc (ZnO,HO), which is readily soluble in excess. The presence of ammoniacal salts prevents the precipitation.

$$ZnO,SO_3 + NH_3 + 2HO = \mathrm{ZnO,HO} + NH_4O,SO_3.$$

If the ammoniacal solution of the oxide be treated with hydrosulphuric acid, the white sulphide (ZnS) is thrown down.

231 (C) *Potash* (KO) behaves in the same manner as ammonia, giving a precipitate (ZnO,HO), soluble in excess. Hydrosulphuric acid throws down the white sulphide from the potash solution.

231*a*. *Identification of metallic zinc.*—Pour a little dilute nitric acid upon a very small piece of metallic zinc in a test-tube; it dissolves with effervescence even in the cold. Prove the presence of zinc in the solution by adding excess of *ammonia* and *hydrosulphuric acid.* (230).

[For the blowpipe tests, see (121, 124)].

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[1] If the yellow hydrosulphate of ammonia be employed, of course any excess of this will impart a yellow appearance to the white precipitate suspended in the liquid.

SECTION IV.

Manganese (Mn).

A solution of sulphate of manganese (MnO,SO_3) may be used.

232. *Hydrosulphuric acid* (HS), when added to an acidified solution (formed by adding a few drops of hydrochloric acid to a little of the solution), gives no precipitate. If the solution is neutral, a partial precipitation of sulphide (MnS) takes place.

233. (C) *Hydrosulphate of ammonia* (NH_4S,HS) gives a flesh-colored precipitate of sulphide of manganese (MnS). If this precipitate be exposed to the air, it is gradually decomposed, and is converted into the dark-brown hydrated sesquoixide ($Mn_2O_3,2HO$), in consequence of the strong affinity of manganese for oxygen, which it absorbs from the air.[1]

234. (C) *Ammonia* (NH_3) throws down a white precipitate of hydrated protoxide of manganese (MnO,HO), which if exposed to the air becomes brown, owing to the formation of the sesquioxide (Mn_2O_32HO), as in the last experiment. $2(MnO,HO)+O=Mn_2O_32HO$.

If chloride of ammonium (NH_4Cl) is present in the solution, it prevents the precipitation of the hydrated protoxide; owing to the formation of double salts of ammonia and manganese, which are not decomposed by ammonia. If the ammoniacal solution be exposed to the air, the brown sesquioxide is gradually precipitated.

235. *Potash* (KO) behaves as ammonia in solutions of manganese.

236. *Carbonate of ammonia* (NH_4O,CO_2) throws down

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[1] Sesquioxide of manganese (Mn_2O_3), manganic acid (MnO_3), and permanganic acid (Mn_2O_7), would not escape detection by hydrosulphate of ammonia, which would convert them, by reduction, into sulphide of manganese. Manganic acid would be known by the green color of its solutions, which changes to the red of permanganic acid on adding a little nitric acid. The red solutions of permanganic acid are bleached instantaneously by hydrosulphuric acid. The solutions of sesquioxide of manganese have a rose color.

a white precipitate of carbonate of manganese (MnO,CO_2), which is less prone to blacken on exposure than the hydrated oxide.

[For the blowpipe tests, see (134, 137).]

SECTION V.

Iron (Fe).

A. *As Protoxide* (FeO) *or some corresponding compound.*

A solution of protosulphate of iron (FeO,SO_3) may be used.

237. On account of the strong tendency of the protoxide of iron to absorb oxygen on exposure to the air, and become sesquioxide, especially in aqueous solutions of its salts, it is difficult to retain the protosalts in solution without some admixture of sesquioxide; so that in testing them, the indications of some of the reagents are frequently more or less different from those caused by a pure protosalt.

238. *Hydrosulphuric acid* (HS) produces no precipitate in acidified solutions of protoxide of iron; a slight precipitation of sulphide (FeS) takes place, however, in neutral solutions of some of its salts, especially when the acid with which it is in combination is a feeble one.

239. *Hydrosulphate of ammonia* (NH_4S,HS), when added to neutral solutions of protoxide of iron, throws down a black precipitate of sulphide (FeS).

$$FeO,SO_3+NH_4S,HS+HO=\mathrm{FeS}+NH_4O,SO_3+\mathrm{HS}.$$

240. (C) *Ammonia* (NH_3) gives a precipitate of hydrated protoxide of iron (FeO,HO), which is at first nearly white, but almost immediately becomes greenish. If this precipitate be exposed to the air, it absorbs oxygen, and is changed into hydrated sesquioxide or peroxide ($2Fe_2O_3,3HO$), which has a reddish-brown or rust color. Chloride and some other salts of ammonium prevent the precipitation of the protoxide by ammonia, forming a solution of a double salt of ammonia and iron, from which the hydrated peroxide is gradually precipitated if exposed to the air.

241. *Nitric acid* (HO,NO_5), on boiling, converts the protoxide of iron (FeO) into sesquioxide (Fe_2O_3), which gives a rust-colored precipitate with ammonia.

$$6FeO+HO,NO_5=3Fe_2O_3+NO_2+HO.$$

242. *Potash* (*KO*) behaves as ammonia.

243. (C) *Ferrocyanide of potassium* ($K_2Fcy+3Aq$)[1] throws down in solutions of protoxide of iron, a precipitate ($KFe3Fcy_2$) which is at first almost white, but rapidly changes to pale blue; the color becomes darker on exposure to the air, owing to the absorption of oxygen, which combines with the potassium and a portion of the iron, forming at the same time Prussian blue, (Fe_4Fcy_3).

244. (C) *Ferridcaynide of potassium* ($K_3,Fdcy$)[2] produces in solutions of the protosalts of iron a beautiful dark-blue precipitate, similar in appearance to Prussian blue, consisting of ferridcyanide of iron (Fe_3Fdcy).

$$K_3Fdcy+3(FeO,SO_3)=Fe_3Fdcy+3(KO,SO_3).$$

[For the blowpipe tests, see (135).]

B. *As Peroxide of Iron* (Fe_2O_3) *or some corresponding compound.*

A solution of the perchloride of iron (Fe_2Cl_3) may be used.

245. *Hydrosulphuric acid* (HS) causes in neutral or acidified solutions of the persalt of iron, a precipitation of sulphur, which gives the solution a milky appearance. This is owing to the decomposition of the hydrosulphuric acid by the peroxide of iron, the hydrogen combining with a portion of its oxygen, reducing it to the state of protoxide, while the liberated sulphur is precipitated in a finely divided state.

$$Fe_2O_3,3SO_3+HS=2(FeO,SO_3)+HO,SO_3+S;$$
$$\text{or } Fe_2Cl_3+HS=2FeCl+HCl+S.$$

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[1] Ferrocyanogen ($Fcy=FeCy_3$), which is here combined with potassium, is a hypothetical radical composed of iron in a peculiar state of combination with cyanogen (C_2N).

[2] Ferridcyanogen ($Fdcy=Fe_2Cy_6$) is a similar hypothetical radical.

246. *Hydrosulphate of ammonia* (NH_4S,HS) separates the whole of the iron from solutions of its persalts, as the black sulphide (FeS), the same compound as that produced in the protosalts. The peroxide (Fe_2O_3) is in fact first converted into the protoxide (FeO) by the deoxidizing affinity of the hydrogen in the hydrosulphate, so that the subsequent change is the same as that produced in the protosulphate (239). If the solution of iron is very dilute, no precipitate appears at first, but the solution becomes green, and if allowed to stand a considerable time, the sulphide gradually separates.

247. (C) *Ammonia* (NH_3) throws down the hydrated peroxide of iron ($2Fe_2O_3,3HO$), in the form of a bulky reddish-brown precipitate, which is insoluble in an excess of ammonia, and is unaffected by the presence of ammoniacal salts.

248. *Potash* (KO) produces the same precipitate, which is insoluble in excess.

249. (C) *Ferrocyanide of potassium* ($K_2Fcy+3Aq$) produces in solutions of the persalts of iron a beautiful precipitate of sesquiferrocyanide of iron, or Prussian blue (Fe_4Fcy_3).

$$3(K_2Fcy)+2Fe_2Cl_3=Fe_4Fcy_3+6KCl.$$

This is an extremely delicate and characteristic test for the persalts of iron, as the precipitate is produced even in very dilute solutions. In testing for iron with ferrocyanide of potassium, however, it must be borne in mind that when added to a solution containing much free acid it is partially decomposed, and a little Prussian blue is formed, even when no iron is present.[1] As the presence of free alkalies also interferes with the formation of the blue precipitate, solutions to be tested with it should be either neutral or slightly acid.

250. *Ferridcyanide of potassium* (K_3Fdcy) produces no precipitate with persalts of iron; it gives, however, a deep green color to the solution.

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[1] This applies especially to mineral acids; in testing such solutions it is advisable that the addition of the ferrocyanide be preceded by that of acetate of potash, so that the mineral acid may be neutralized, and acetic acid liberated in its stead.

250*a*. *Identification of metallic iron.*—Pour some dilute nitric acid upon two or three iron filings; accelerate the solution by heat, and prove the presence of peroxide of iron by testing with ammonia (247).

SECTION VI.

Nickel (Ni).

A solution of the sulphate of nickel (NiO,SO_3) may be used.

251. *Hydrosulphuric acid* (HS) causes no precipitate in acidified solutions of nickel; but if the solution is neutral, especially if the acid of the salt be a feeble one, a partial precipitation of sulphide of nickel (NiS) takes place.

252. *Hydrosulphate of ammonia* (NH_4S,HS) throws down, from neutral solutions, a black precipitate of sulphide (NiS) which is slightly soluble in excess,[1] giving a brownish tint to the solution. Sulphide of nickel is sparingly soluble in dilute hydrochloric acid, but dissolves immediately on addition of nitric acid.

253. (C) *Ammonia* (NH_3) causes a pale green precipitate of hydrated oxide of nickel (NiO,HO) which redissolves when the ammonia is added in excess, owing to the formation of ammoniated oxide of nickel, and of a double salt of ammonia and nickel (NH_4O,SO_3,NiO,SO_3), which are soluble in water. If potash (KO) be added to the ammoniacal solution, it precipitates the hydrated oxide of nickel. The presence of ammoniacal salts in the nickel solution prevents the precipitation by ammonia.

254. *Potash* (KO) also throws down the hydrated oxide of nickel (NiO,HO), which is insoluble in an excess of potash.

255. *Carbonate of Ammonia* (NH_4O,CO_2) produces a

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[1] That is, in excess of the ordinary yellow hydrosulphate: the colorless freshly prepared hydrosulphate of ammonia does not dissolve it.

precipitate of carbonate of nickel (NiO,CO_2), which redissolves in excess.

256. (C) *Cyanide of potassium* (*KCy*), throws down a precipitate of cyanide of nickel ($NiCy$), which has a yellowish-green color: it redissolves in an excess of the alkaline cyanide, forming a dull yellow solution of the double cyanide of nickel and potassium (*$NiCy,KCy$*), from which the cyanide of nickel is again precipitated on the addition of dilute hydrochloric acid. If the precipitate be boiled with potash, it is converted into oxide of nickel, cyanide of potassium being formed.

257. *Ferridcyanide of potassium* (K_3Fdcy) produces a greenish yellow precipitate of ferridcyanide of nickel (Ni_3Fdcy).

[For the blowpipe test, see (136).]

SECTION VII.

Cobalt (Co).

A solution of the nitrate (*CoO,NO_5*) may be used.

258. *Hydrosulphuric acid* (HS) gives in acidified solutions no precipitate. If the solution is neutral, a slight precipitation of the black sulphide of cobalt (CoS) takes place.

259. *Hydrosulphate of ammonia* (*NH_4S,HS*) throws down from neutral solutions a black precipitate of the sulphide (CoS), which is insoluble in excess, and also in hydrochloric acid.

260. (C) *Potash* (*KO*) throws down a precipitate of a blue color, consisting of a basic salt of cobalt, which soon becomes greenish if exposed to the air, owing to the absorption of oxygen; and lastly, especially if the solution be boiled, dirty red, owing to the formation of hydrated oxide of cobalt (CoO,HO). The precipitate is insoluble in excess of potash.

261. (C) *Ammonia* (*NH_3*) behaves as potash, but the precipitate readily dissolves in an excess, forming double

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[1] Cyanogen (Cy), which is here combined with potassium, is a compound of carbon and nitrogen (C_2N).

salts of cobalt and ammonia, which are soluble in water. If the red ammoniacal solution is exposed to the air, it gradually becomes darker, owing to the absorption of oxygen, and formation of peroxide of cobalt (Co_2O_3).

262. *Carbonate of ammonia* ($NH_4O.CO_2$) gives a precipitate of carbonate of cobalt (CoO,CO_2), which readily dissolves in excess of the precipitant, yielding a red solution.

263. (C) *Cyanide of potassium* (KCy) when added to a solution of cobalt gives a pale brown precipitate of cyanide of cobalt ($CoCy$), which when heated with an excess of cyanide of potassium, readily redissolves, forming cobalticyanide of potassium ($K_3Co_2Cy_6$). The addition of hydrochloric acid causes no precipitate in this solution (256).[1]

264. (C) *Ferridcyanide of potassium* (K_3Fdcy) gives a purple-brown precipitate (Co_3Fdcy), or in very dilute solutions, a brown coloration.

[For the blowpipe test, see (133).]

Summary of Class III.

265. In order to ascertain whether a solution under examination contains a metal belonging to this class, acidulate it with hydrochloric acid and add hydrosulphuric acid; if all metals belonging to Class IV. be absent, there will be no precipitate except, perhaps, of sulphur, which may be recognized by its imparting a milkiness to the liquid instead of separating as a distinct precipitate. On adding, to another portion of the solution, chloride of ammonium,[2] ammonia, and hydrosulphate of ammonia, the metals of Class III. will be precipitated, and may be distinguished to some extent by the color of the precipitate. It is not advisable to attach much importance to the particular hue of the precipitate, since it is so easily modified by trifling causes (229); if the precipitate be decidedly black, however, the presence of iron, cobalt, or nickel, is certain. To distinguish between them, add potash to a fresh portion of the solution; a dingy green precipitate,

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[1] In order to insure the complete conversion of the cobalt into cobalticyanide of potassium, the solution should be boiled for a minute or two.

[2] To prevent the precipitation of magnesia (209).

changing to rust-color by exposure to air, indicates iron as protoxide (FeO); a rust-colored precipitate indicates iron as sesquioxide (Fe_2O_3); an apple-green precipitate indicates nickel; and a blue precipitate becoming dirty red when boiled, cobalt. On the other hand, if the precipitate produced by hydrosulphate of ammonia had any other color than black, the metal present would be either manganese, zinc, chromium, or aluminum.[1] To distinguish between them; add potash in excess to a fresh portion of the solution; a white precipitate insoluble in excess, and rendered brown by exposure, indicates manganese; if there be no precipitate, boil the solution, when chromium would be precipitated as green oxide; test another part of the solution with acetate of lead for chromic acid (227). If these give no indication, aluminum or zinc must be present. Add excess of ammonia to a fresh portion of the solution; a transparent gelatinous precipitate indicates aluminum; if there be no precipitate, zinc must be present, and the addition of hydrosulphuric acid will produce a white precipitate in the ammoniacal solution.

265*a*. If iron be present in combination with cyanogen, forming a ferrocyanide or a ferridcyanide, it may escape detection by the ordinary tests, unless the cyanogen be decomposed by ignition or otherwise. The addition of protosulphate or perchloride of iron would at once indicate the presence of ferro- or ferridcyanogen (243, 244, 249, 250).

CHAPTER V.

METALS BELONGING TO CLASS IV.

Arsenic, Antimony, Mercury, Lead, Copper, Silver, Tin and Bismuth.

266. These metals are distinguished from those of the three preceding classes, by being precipitated from their

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[1] **The phosphates and oxalates of the alkaline earths would also be precipitated here: they are white, and would be distinguished from alumina and oxide of zinc by their insolubility in potash.**

acidified solutions when treated with hydrosulphuric acid. It is remarkable that nearly all the metals the compounds of which are most eminently poisonous belong to this class; and as these are the most important, especially to the medical student, they are placed first, and described in the order of their importance.

SECTION I.

Arsenic (As).

A. *As Arsenious Acid* (AsO_3).

267. On account of the highly poisonous nature of arsenic, great care should be taken, in the following experiments, not to use more than is absolutely necessary to exhibit its peculiarities. In all these experiments (except those of solution), a fragment the size of a small pin's head is quite sufficient. There is also another advantage in using such small quantities—namely, that in most medico-legal investigations the quantity to be looked for is very minute, and it is consequently very important that the student should make himself familiar with the appearances which would, under these circumstances, present themselves.

268.[1] (C) If a small fragment of arsenious acid be heated on charcoal before the blowpipe, it is wholly volatilized, and a smell of garlic will generally be perceptible, especially when it is subjected to the reducing flame. Both metallic arsenic and its oxide are volatile when heated, but the fumes of the latter have no smell. The odor of arsenic vapor appears to be due to the metal while undergoing oxidation, and may be caused perhaps by the formation of a lower oxide than the arsenious acid; it is always observable when metallic arsenic is volatilized in contact with the air.

269. (C) Place a fragment of arsenious acid in a narrow tube (99), warm the upper part of the tube by drawing it once or twice through a gas or spirit flame, and apply a gentle heat to the arsenious acid. It sublimes

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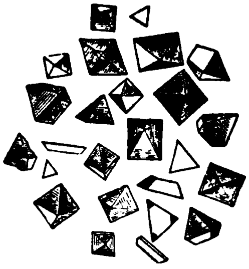
[1] **It has been thought advisable to repeat the blowpipe tests for arsenic (already described in Part I.), in order to exhibit in this section all the leading characters of that important poison.**

Fig. 64.

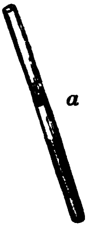


Crystals of Arsenious Acid.

without decomposition, and condenses in the cool part of the tube, in the form of minute sparkling octohedral crystals (Fig. 64), which should be examined with a lens, as they are highly characteristic. The size and regularity of the crystals depend on the slowness with which the vapor is condensed. If the surface of the glass on which the condensation takes place is quite cold, the sublimate is often amorphous.[1]

270. (C) Mix a little arsenious acid (AsO_3) with black flux, which if at all damp should be previously dried on the sand-bath, and heat a little of the mixture in a clean tube before the blowpipe (105, 106). The arsenious acid is deoxidized by the carbon of the flux, and the metallic arsenic thus reduced sublimes, and condenses in the upper part of the tube, forming a more or less brilliant metallic crust, *a* (Fig. 65). $AsO_3 + 3C = As + 3CO$.

Fig. 65.



Crust of Arsenic.

If a moderate heat be now applied to the sublimate, it will again volatilize, and if any of the vapor escapes from the tube, it may be recognized by its characteristic odor of garlic (268).

271. (C) Cut off, by means of a file, the portion of the tube containing the crust, wrap it in a piece of paper, break it into fragments, and place some of them in another tube. Sublime the arsenic by a

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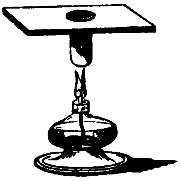

[1] Dr. Guy has devised a very simple method of obtaining this sublimate, and others of a similar kind, upon a flat slide for microscopical examination. In a flat plate of metal or porcelain, a hole is drilled, sufficiently large to receive a very short test-tube, about an inch and a half long, and half an inch in diameter; the arsenious acid is gently heated in this tube over a spirit-lamp flame, and the crystalline sublimate is allowed to deposit upon a glass slide lightly warmed and placed over the mouth of the tube.

gentle heat in the tube, and observe the conversion of the metal into crystalline arsenious acid, which is formed by the action of the atmospheric oxygen contained in the tube.

272. Boil a few grains of arsenious acid with water (in which it is sparingly soluble), in a flask: filter the solution from the undissolved portion, and retain it for testing.

273. (C) *Hydrosulphuric acid* (HS) gas,[1] when passed through a solution of arsenious acid, gives it a bright yellow color, due to the formation of tersulphide of arsenic; $AsO_3+3HS=AsS_3+3HO$.

If the solution be acidified, however, with a few drops of hydrochloric acid, a much more rapid and complete decomposition takes place; and if the gas be passed through the solution for some time, a complete separation of the arsenic may in this way be effected. The sulphide of arsenic thus formed has a bright yellow color.

274. (C) Pour a little of the solution containing the yellow precipitate into a test-tube, and add a few drops of ammonia, which will very easily dissolve it. (Sulphide of cadmium, which much resembles the sulphide of arsenic, is insoluble in ammonia.)

275. (C) Collect the sulphide of arsenic upon a small filter (66), and wash it twice or thrice with water; take a little of it upon the end of a glass rod, and mix it with a few drops of water in a test-tube; add a little solution of sesquicarbonate of ammonia, heat gently, and observe that the sulphide of arsenic readily dissolves.

276. (C) Repeat the experiment with hydrosulphate of ammonia, which will dissolve it even more readily.

277. (C) Dry the remainder of the sulphide of arsenic very carefully (631), mix a little of it with black flux, or with dried carbonate of soda and a little charcoal (270), and heat it in a tube; observe the formation of a metallic crust of arsenic, and identify it as in (271).

$$AsS_3+3(KO,CO_2)+C_3=As+3KS+3CO_2+3CO.$$

278. A very excellent method of testing the sulphide of arsenic was proposed by Fresenius: it consists in re-

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[1] For the preparation of hydrosulphuric acid, see (687).

ducing the sulphide to the metallic state by fusion with carbonate of soda and cyanide of potassium, and carrying the vapor of the metal, by means of a gentle stream of carbonic acid, into the narrow point of a glass tube, when the smallest quantity becomes visible. The figure will show the arrangement adopted for this purpose. The larger flask contains fragments of marble covered with water, to which hydrochloric acid is added through the funnel tube for the purpose of evolving carbonic acid, which is dried by passing through oil of vitriol in the smaller flask. The sulphide of arsenic is mixed with about 12 parts of a mixture of 3 parts of carbonate of soda and 1 part of cyanide of potassium (previously well dried); the mixture is introduced into the tube[1] at *d* by means of a paper gutter (p. 79), and the tube is then attached to the apparatus. As soon as all the air has been expelled, and the carbonic acid is passing through the oil of vitriol at the rate of about a bubble in a second, the mixture is thoroughly dried by gently heating the tube throughout its whole length with a spirit lamp; as soon as all the moisture is expelled, the shoulder is so heated that no arsenic may condense there, and is kept hot while a second spirit lamp is employed to heat the mixture; the heat may be ultimately increased by the blowpipe, if

Fig. 66.

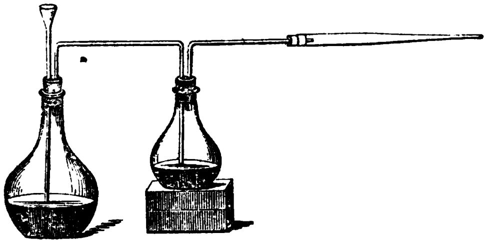
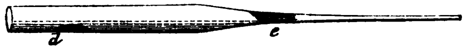


Fig. 67.



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[1] The tube is made of stout German glass.

necessary; the metallic arsenic condenses in the narrow part of the tube, at *e*, and any of the tests may be applied in order to complete its identification.

Antimony gives no deposit when its sulphide is treated in this manner.

279. *Hydrosulphate of ammonia* (NH_4S,HS), when added to a solution of arsenious acid, also causes the formation of the yellow sulphide (AsS_3), which however does not precipitate, but remains dissolved as the double sulphide of arsenic and ammonium (NH_4S,AsS_3). If hydrochloric acid be added in excess to the mixture, the sulphide of arsenic is immediately precipitated, of a somewhat lighter color than that thrown down by hydrosulphuric acid, owing to the admixture of a little sulphur derived from the hydrosulphate of ammonia.

280. (C) *Ammonio-nitrate of silver*[1] ($AgO,NO_5,2NH_3$) throws down, in solution of arsenious acid, a canary-colored precipitate of arsenite of silver ($3AgO,AsO_3$), which is soluble both in ammonia and nitric acid. It must be remembered that *phosphate of soda* also produces, with nitrate of silver, a similar precipitate, which is equally soluble in nitric acid and ammonia.

A solution of arsenious acid in water gives no precipitate with *nitrate of silver* unless ammonia is added, but this must be done very carefully, as a very slight excess will redissolve the yellow precipitate.

281. (C) *Ammonio-sulphate of copper*[2] ($CuO,SO_3,2NH_3,HO$) produces in solution of arsenious acid, a delicate green precipitate of arsenite of copper ($2CuO,HO,AsO_3$), which dissolves readily both in ammonia and nitric acid. It must be borne in mind, in employing this test, that a similar precipitate is produced when the solution of copper is added to liquids containing some vegetable substances, though no arsenic may be present.

Marsh's Test.

282. (C) It is well known that when zinc is treated with dilute sulphuric acid, it is oxidized at the expense

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For the preparation of this reagent, see (737).
For the mode of preparing it, see (733).

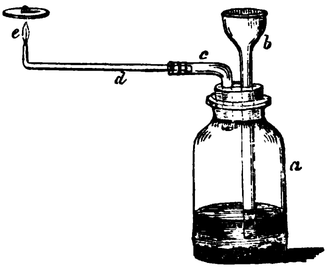
of the oxygen of the water, and hydrogen gas is given off (11). If in addition to the zinc and dilute sulphuric acid, either of the oxides of arsenic be present, the zinc abstracts oxygen from it as well as from the water; and the metallic arsenic thus formed, combines, at the moment of its liberation, with some of the hydrogen simultaneously produced, and forms a gaseous compound called *arseniuretted hydrogen* (AsH_3), which passes off mixed with the excess of hydrogen.[1]

$$AsO_3 + 6Zn + 6(HO,SO_3) = 6(ZnO,SO_3) + AsH_3 + 3HO.$$

Now if this arseniuretted hydrogen is heated strongly, either by burning in the air, or by passing through a red-hot tube, it is decomposed, and metallic arsenic or its oxide is deposited in the solid state.

283. Several forms of apparatus have been contrived for making use of this property in the detection of arsenic;—of these the following is in practice the most convenient: The bottle *a* (Fig. 68) should be capable of containing six or eight ounces of water, and is connected by means of a perforated cork with the tubes *b* and *c*, which should be about half an inch in diameter; to the latter is attached, by means of a cork, the tube *d*, which should be made of hard German glass, bent at a right angle, having the end *e* drawn off so as to diminish the aperture. A few fragments of zinc are placed in the bottle and covered with water; and when the cork with its tubes is attached, pour a little dilute sulphuric acid down the tube *b*, which should reach nearly to the bottom of the bottle, and allow the gas (hy-

Fig. 68.



Marsh's Test.

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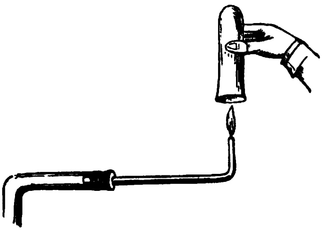
[1] It must be borne in mind that this gas, like most of the other compounds of arsenic, is highly poisonous; so that the experiment should never be performed in a close room, but in the open air or in a well-ventilated apartment.

drogen) to be given off for five minutes.[1] Then kindle the hydrogen at the mouth of the tube, and depress a porcelain plate (the lid of a Berlin crucible) upon the flame for a second or two; observe whether any brown stain of arsenic is deposited upon it. This precaution is necessary, since some kinds of sulphuric acid, and also of zinc, contain traces of arsenic. If no stain is produced, it may be assumed that the materials are pure.

284. The solution containing (or suspected to contain) arsenic, is now introduced through the tube *b*. If arsenic is present in the liquid, it will cause the formation of arseniuretted hydrogen, which will impart a livid hue to the flame of the hydrogen; on depressing the porcelain plate again upon the flame, the metallic arsenic will be deposited in the form of a dark shining spot. By applying heat to the dark spots, they are readily volatilized, and the fumes will be found to have the characteristic odor of garlic.

A few of these spots should be retained for further examination, and for comparison with those of antimony.

Fig. 69.



285. Hold a short test-tube with the mouth downwards, just above the apex of the flame (Fig. 69) for a few moments, so as to collect some of the arsenious acid formed by the oxidation of the arsenious vapor, and reserve the tube for comparison with antimony.

286. A deposit of metallic arsenic may also be obtained by strongly heating the tube through which the gas is passing, at the point *d* (Fig. 68), when a lustrous mirror will be formed, not exactly at the heated point, but a quarter or half an inch beyond (in consequence of

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[1] The reason why it is not safe to apply the light at once, is that a mixture of hydrogen and common air is highly explosive (12), so that it is necessary to allow time for the whole of the common air to be expelled by the hydrogen; as otherwise serious injury might be caused by an explosion of the mixed gases.

the volatility of arsenic). The portion of the tube which contains the deposit may be filed off and examined as directed at (271).

287. Marsh's test as just described is so extremely delicate, that it is capable of detecting arsenic in a solution containing the millionth of its weight of the acid, and may be considered the most conclusive test which we possess. It is however liable to this objection, which is, in practice, easily overcome. It is found that antimony, when present in a mixture of zinc and dilute sulphuric acid, combines with the liberated hydrogen, precisely in the same way as arsenic, forming an analogous compound called antimoniuretted hydrogen (SbH_3); which, when heated, is decomposed, and the metallic antimony is at the same time deposited. Hence it is extremely important that we should be able to distinguish accurately between them, as otherwise we should not be sure whether the crusts produced by Marsh's test were due to arsenic or antimony. One or two experiments are generally sufficient to enable us to do this.

288. For the purpose of comparison, empty the zinc and sulphuric acid from the bottle used for the arsenic experiments (283), wash it well, and substitute fresh zinc and acid. When the gas has been coming off about five minutes (Note to 283), pour in a few drops of a solution of the double tartrate of antimony and potash (tartar emetic $KO,SbO_3,C_8H_4O_{10}+Aq$), and apply heat as before at the point *d*. A crust of antimony will be deposited *at the heated point*, and not, as in the case of arsenic, at a little distance from it; this is owing to the antimony being less volatile than arsenic, and it would be found impossible to volatilize it by the heat of a common spirit lamp. In this respect, therefore, we are enabled in some measure to judge whether the stain is due to antimony or arsenic.

289. Light the jet of gas that issues from the aperture *e* as in (283), and hold over the flame a porcelain plate as before: a deposit of metallic antimony will be formed similar to that of arsenic, but blacker and less shining.

Prepare a few of these spots for comparison with those of arsenic formed in (283).

290. (C) Apply the heat of a spirit lamp to one of each

kind of spot, and observe the superior volatility of the arsenic, and the garlic odor of its vapor.

291. (C) Moisten one of each kind of spot with yellow hydrosulphate of ammonia (NH_4S,HS) on the end of a glass rod, and observe that the antimony *is immediately dissolved*, while the arsenic remains nearly unaffected for a considerable length of time. This is a most valuable means of distinguishing between them, and was first observed by Dr. Guy.

292. (C) If the spots be moistened with a solution of chloride of lime ($CaO,ClO+CaCl$), the arsenic will dissolve, while the antimony will remain unaffected.

293. (C) The following may also be taken as a distinguishing test between arsenic and antimony when Marsh's process is followed. Light the jet of gas issuing from the apparatus, and hold over it a short tube as in (285), so as to collect a little of the oxide of antimony (SbO_3) formed by the oxidation of the antimonial vapor. When the tube is cold, pour in a few drops of water, and boil; treat the arsenious acid in the same way: observe that the latter dissolves in the water, while the oxide of antimony remains insoluble. The solution of arsenious acid may then be divided into three portions and tested; the first with *hydrochloric* and *hydrosulphuric acid* (273); the second with *ammonio-nitrate of silver* (280); and the third with *ammonio-sulphate of copper* (281).

These experiments, in conjunction with the other liquid tests, will be found sufficient to prevent the possibility of error in the use of Marsh's test.

Reinsch's Test.

294. (C) This test is founded on the circumstance that when a metal, such as copper, is heated in a solution of another metal more electro-negative than itself, the latter is separated in the metallic state, and deposited on the surface of the former, which is at the same time dissolved in equivalent proportion. A little of the solution containing arsenic is acidified with a few drops of hydrochloric acid, and boiled in a test-tube with three or four strips of clean copper foil: the arsenic, being more elec-

tro-negative than the copper, is deposited on the surface of the foil, and the whole is in this way separated from the solution.

$$AsO_3+3Cu=As+3CuO.$$

295. The appearance of a metallic deposit on the copper is not, however, necessarily a proof of the presence of arsenic, since other metals (as bismuth, silver, mercury, or antimony) would produce a similar incrustation, being all more electro-negative than copper. Arsenic, however, is readily distinguished from any of these in the following manner:—

296. Take the copper strips out of the solution, and dry them cautiously between folds of filtering paper: place them in a small tube closed at one end, and apply heat, when the arsenic will be volatilized, and, becoming oxidized by the air contained in the tube, will form a crystalline sublimate in the upper part (269.)

297. Had the deposit on the copper consisted of silver, it would not have been volatilized when heated: if it were mercury, minute globules of the metal would have condensed in the cool part of the tube: and had it been bismuth or antimony, a higher degree of heat would have been necessary to sublime it; the sublimate would have been white and amorphous instead of crystalline;—and when treated with water, would prove insoluble, while the arsenious acid would dissolve, and the solution, on being tested, would show the presence of arsenic.[1]

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[1] If the arsenic be present in the shape of arsenic acid, the copper separates it with greater difficulty, and it is necessary to add a very large proportion of hydrochloric acid. A great obstacle to the use of this simple and delicate test is the difficulty of obtaining hydrochloric acid, and especially copper, perfectly free from arsenic. In testing these, a quantity of hydrochloric acid equal to that employed in testing the suspected substance, should be diluted with four times its bulk of water, boiled in a small flask, and a small strip of the copper introduced; if after boiling for 15 or 20 minutes, there is no deposit upon the copper, the hydrochloric acid may be considered sufficiently pure. Several of the strips of copper should be strongly heated in a small closed tube to prove that they will not afford crystals of arsenious acid.

No oxidizing agent (such as nitric or chloric acid) must be present in the liquid to be tested, or the copper will be dissolved.

B. *As Arsenic Acid* (AsO_5).

298. Mix a little arsenious acid with nitre (KO,NO_5), and heat it in a tube. The nitric acid of the nitre gives up a portion of its oxygen to the arsenic, forming arsenic acid (AsO_5).

$$2AsO_3+2NO_5=2AsO_5+2NO_3.$$

Dissolve the fused mass in water, and test it with *nitrate of silver* (AgO,NO_5): a reddish-brown precipitate of arseniate of silver ($3AgO,AsO_5$) is thrown down, which is soluble in nitric acid, and also in ammonia.

299. Arsenic acid, even when mixed with hydrochloric acid, is not precipitated by hydrosulphuric acid unless the solution is boiled, or allowed to stand for some time.

Detection of Arsenic in Organic Mixtures.

300. In most cases of medico-legal investigation as to the presence of arsenic, we have to deal with mixtures containing a considerable quantity of organic matter, both liquid and solid, which seriously interferes with the action of the tests. Several methods have been employed to get rid of these matters, but the following is perhaps the simplest, and at the same time the most effectual: it is a modification of Reinsch's test.

If the organic mixture suspected to contain arsenic is fluid, it is, previous to filtration, boiled for half an hour with about one-tenth of its bulk of strong hydrochloric acid, the purity of which should of course be ascertained (see note to 297); and if necessary, filtered from any solid matter. It is then boiled with copper foil, when the arsenic, if present, is deposited on the copper, which must be subsequently heated in a tube, according to the directions already given (296).

301. If the matter to be examined is solid, it is treated with dilute hydrochloric acid containing about one-tenth of the strong acid, boiled for half an hour or an hour, filtered if necessary, and then boiled with copper as before.

302. If the arsenic is present only in very small quantity, a quarter of an hour may elapse before the deposi-

tion takes place; and if it does not then appear, the boiling should be continued half an hour or even longer, before we finally conclude that no arsenic is present.

303. Should this test, when applied to a small portion of the substance, indicate the presence of arsenic, the operator should endeavor to obtain the metal in the form of sulphide. This is attended with considerable difficulty in such mixtures as the contents of a stomach, on account of the heterogeneous nature of the organic matters. The following method is generally attended with satisfactory results: The mixture is heated on a water-bath, with about a fourth of its bulk of hydrochloric acid, and chlorate of potash is added in small portions, till the solid matter is disintegrated and the liquid is no longer viscid; the heat is continued till the odor of chlorine has nearly disappeared, and the mixture is then filtered; it is afterwards diluted with an equal bulk of water, thoroughly saturated with sulphuretted hydrogen, heated to boiling, again saturated with sulphuretted hydrogen, and set aside in a loosely stopped flask for two or three hours, in a warm situation. This method has the advantage of permitting the detection of other metals of the fourth class, and allows the liquid to be afterwards tested for zinc.

For further particulars on this subject, the student may refer to Dr. Christison's Treatise on Poisons, or to Dr. Guy's excellent work on Forensic Medicine.

SECTION II.

Antimony (Sb).

304. Add *dilute hydrochloric acid*, drop by drop, to solution of tartar-emetic ($KO,SbO_3,C_8H_4O_{10}+Aq$); a white precipitate of oxide of antimony (SbO_3) will be produced at first, but will be easily dissolved by the acid.

305. (C) To this solution (containing only just enough acid to redissolve the precipitate) add eight or ten times its volume of *water*, when it will become milky from the separation of oxychloride of antimony ($SbCl_3,2SbO_3$), but may be rendered clear again by adding tartaric acid.

306. (C) Acidify a solution of tartar-emetic with hydro-

chloric acid, and add *hydrosulphuric acid*, which will produce an orange precipitate of tersulphide of antimony (SbS_3).[1]

307. (C) Collect the tersulphide of antimony upon a small filter, wash it once or twice with water, perforate the filter with a glass rod and wash the precipitate into a test-tube, using as little water as possible. Add sesquicarbonate of ammonia and heat gently; the sulphide of antimony dissolves only to a very slight extent, and is thus distinguished from sulphide of arsenic (275). Add a little hydrosulphate of ammonia, and the precipitate will readily dissolve, but may be precipitated, mixed with a little sulphur, by dilute hydrochloric acid.

307*a*. (C) Add *sesquicarbonate of ammonia* to solution of terchloride of antimony. A white precipitate of hydrated teroxide of antimony is produced, which dissolves almost entirely on boiling with an excess of the carbonate of ammonia.

308. (C) When a solution containing antimony is brought in contact with metallic zinc and dilute sulphuric acid, the antimony is reduced and combines with the hydrogen, forming antimoniuretted hydrogen (SbH_3), which is decomposed when burnt, or when passed through a red-hot tube, with the formation of a deposit of metallic antimony. This mode of detecting antimony has already been described (288).

309. *Identification of metallic antimony.*—Boil a little powdered antimony with dilute nitric acid, which will convert it into a white powder (hydrated antimonic acid $SbO_5,4HO$). Boil another portion of the metal with hydrochloric acid, and add nitric acid, very carefully, drop by drop, till the antimony is dissolved, as terchloride ($SbCl_3$). Prove the presence of antimony in the solution by largely diluting one portion (305), and by testing another with hydrosulphuric acid (306).

[For the blowpipe test for antimony, see (119).]

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[1] Antimonic acid (SbO_5) would furnish a similar precipitate.

SECTION III.

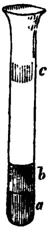
Mercury (Hg).

A. *As suboxide* (Hg_2O) *or some corresponding compound.*

310.[1] (C) Heat a small fragment of calomel (not larger than a small pin's head) in a clean tube. It becomes pale yellow, and, being volatile, it sublimes, and condenses in the upper part of the tube; on cooling, the color disappears.

311. (C) Mix a little dry carbonate of soda (NaO,CO_2) with a very minute quantity of calomel, and put the mixture into a tube *a* (Fig. 70); then cover it with a layer of carbonate of soda in powder, about a quarter of an inch deep, *b*, and apply heat (105). The calomel is decomposed, and minute globules of metallic mercury condense in the cool part of the tube at *c*.

Fig. 70.



$$\mathrm{Hg_2Cl+NaO,CO_2=NaCl+}\mathit{Hg}_2\mathrm{+O+CO_2}.$$

312. Boil a little calomel with distilled water in a test-tube; filter the water into another tube, and test it with hydrosulphate of ammonia; no effect is produced, proving that the calomel is insoluble in water.[2]

313. (C) *Potash* (*KO*) or *ammonia* (NH_3) poured on the calomel, decomposes it, turning it black, owing to the formation of the suboxide (Hg_2O). Chloride of potassium or ammonium is at the same time formed.

$$\mathrm{Hg_2Cl+}\mathit{KO}\mathrm{=Hg_2O+}\mathit{KCl}.$$

314. (C) *Hydrochloric acid* (*HCl*), added to a solution of suboxide of mercury (*e. g.*, the protonitrate Hg_2O,NO_5), throws down a white precipitate of subchloride (calomel, Hg_2Cl), which is insoluble in excess.

$$\mathit{Hg_2O,NO_5+HCl}\mathrm{=Hg_2Cl+}\mathit{HO,NO_5}.$$

Ammonia converts the precipitate into black suboxide of mercury (Hg_2O).

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[1] The blowpipe tests for calomel are repeated here, on account of its special importance.

[2] If the calomel contained any corrosive sublimate, the hydrosulphate would produce a black precipitate.

315. (C) Place a strip of clean *copper* in the mercurial solution, and observe the deposition of metallic mercury.

$$Hg_2O,NO_5+\mathrm{Cu}=Hg_2+CuO,NO_5.$$

B. *As Oxide of Mercury* (HgO) *or some corresponding compound.*

The perchloride of mercury or corrosive sublimate (HgCl), either solid or in solution, may be used.

316. (C) Heat a small fragment of the perchloride in a tube; it fuses, boils, and sublimes into the upper part of the tube (104).

317. (C) Repeat the experiment described in (311), using a very small particle of the perchloride instead of calomel; metallic mercury sublimes in both cases.

318. Boil a little with water, in which it readily dissolves, thus differing from the subchloride.

319. (C) Test a solution of the perchloride with a small quantity of *hydrosulphuric acid* (HS). A white precipitate is first formed, which, on the addition of more of the precipitant, gradually becomes darker, and ultimately black. This change of color is owing to the formation first of a double compound of sulphide and chloride of mercury (2HgS,HgCl), which is white; and when the hydrosulphuric acid is added in excess, the whole of the mercury is converted into the black persulphide (HgS). The precipitate is insoluble in hydrochloric and nitric acids separately, but is readily dissolved by a mixture of the two, and again converted into the perchloride.

320. *Ammonia* (NH_3) throws down a white precipitate of amidochloride of mercury.[1]

321. (C) *Potash* (KO) gives a yellow precipitate of oxide of mercury (HgO), which is insoluble in excess. If ammoniacal salts are present, the precipitate formed by potash is white, and consists of the same compound as that thrown down by ammonia (320).

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[1] *White precipitate* may be regarded as a chloride of dimercurammonium, that is, as chloride of ammonium NH_4Cl, in which half the hydrogen is displaced by mercury (NH_2Hg_2Cl.)

322. (C) When *protochloride of tin* ($SnCl$) is added in small quantity, the perchloride is reduced to the state of subchloride ($\mathrm{Hg_2Cl}$), which separates as a white precipitate. If the salt of tin be added in excess, and the mixture boiled, the mercury is reduced to the metallic state.

$$HgCl+SnCl=Hg+SnCl_2.$$

323. (C) *Iodide of potassium* (KI) causes a most beautiful red precipitate of periodide of mercury (HgI), which surpasses even vermilion in brilliancy of color. It is readily soluble in an excess of either of the solutions.

$$HgCl+KI=\mathrm{HgI}+KCl.$$

324. (C) If a solution containing mercury be acidified with hydrochloric acid, and boiled with a few strips of clean copper, the surface of the latter becomes coated with a lustrous film of metallic mercury, and if the slips be dried upon blotting paper and heated in a small tube closed at one end, globules of mercury may be obtained.

SECTION IV.

Lead (Pb).

A solution of the acetate ($PbO,C_4H_3O_3+3\,\mathrm{Aq}$) or the nitrate ($PbO,NO_5$) may be used.

325. *Hydrosulphuric acid* (HS) throws down, in solutions containing lead, a dense black precipitate of sulphide of lead (PbS).

$$PbO,NO_5+\mathrm{HS}=\mathrm{PbS}+HO,NO_5.$$

If the sulphide be boiled with dilute nitric acid, a little of it is converted into the insoluble sulphate ($\mathrm{PbO,SO_3}$), but the greater part is dissolved.

326. *Ammonia* (NH_3) and *Potash* (KO) throw down white precipitates, consisting of oxide of lead in combination with a small quantity of acid (basic salts). The precipitate by potash is soluble in excess.

Ammonia produces scarcely any precipitate in a solution of acetate of lead owing to the formation of the subacetate ($3PbO,C_4H_3O_3$), which is soluble.

327. (C) *Sulphuric acid* (HO,SO_3) produces a white precipitate of sulphate of lead (PbO,SO_3), which is sparingly soluble in acids, but soluble in potash, and also in acetate of ammonia ($NH_4O,C_4H_3O_3$).

$$PbO,NO_5+HO,SO_3=PbO,SO_3+HO,NO_5.$$

If the precipitate be moistened with a solution of hydrosulphate of ammonia, it is instantly blackened, owing to the formation of sulphide of lead (PbS): it is distinguished in this way from the insoluble sulphates of baryta and strontia.

328. (C) *Hydrochloric acid* (HCl) throws down a white and often crystalline precipitate of chloride of lead ($PbCl$). If the solution with the precipitate be boiled, a portion of the chloride dissolves, and is deposited again on cooling, in the form of needle-shaped crystals. If the solution of lead is dilute, the chloride does not precipitate, as it is somewhat soluble in water.

$$PbO,NO_5+HCl=PbCl+HO,NO_5.$$

The chloride of lead is not dissolved by an excess of ammonia.

329. (C) *Chromate of Potash* (KO,CrO_3) gives a fine yellow precipitate of chromate of lead (PbO,CrO_3), which is insoluble in dilute acids, but soluble in potash. This substance is the basis of the pigment known in commerce as chrome yellow.

330. (C) *Iodide of potassium* (KI) also gives a beautiful yellow precipitate of iodide of lead (PbI), which is rather lighter in tint than the chromate. If the iodide thus formed be boiled with water, it dissolves and again separates on cooling, in the form of brilliant crystalline scales, which are extremely beautiful.

331. *Identification of metallic lead.*—Boil a small piece of lead (*e.g.*, a shot) with a little dilute nitric adid, which will gradually dissolve it; test different portions of the solution with sulphuric acid (327), and hydrochloric acid (328). Strong nitric acid will not easily dissolve lead, the metal becoming coated with nitrate of lead, which is not soluble in the strong acid.

[For the blowpipe test, see (115).]

SECTION V.

Copper (Cu).

A solution of sulphate of copper (CuO,SO_3) may be used.

332. *Hydrosulphuric acid* (HS) *and hydrosulphate of ammonia* (NH_4S,HS) throw down a black precipitate of sulphide of copper (CuS) from solutions of copper salts, whether neutral, acid, or alkaline.

$$CuO,SO_3+\mathrm{HS}=\mathrm{CuS}+HO,SO_3.$$

333. (C) *Ammonia* (NH_3), when added in small quantity, throws down a pale blue precipitate consisting of a basic salt of copper which immediately redissolves when the ammonia is added in excess; the solution thus formed has a beautiful deep blue color owing to the formation of the ammonio-sulphate of copper ($\mathrm{CuO,SO_32NH_3,HO}$.)

334. (C) *Potash* (KO) produces in cold solutions of copper, a pale blue precipitate of hydrated oxide (CuO,HO). If the mixture be boiled, or if the potash be added to a hot solution, the precipitate becomes black, owing to the decomposition of the hydrated oxide at a temperature of 212°, and formation of the anhydrous black oxide (CuO). The potash must for this purpose be added slightly in excess, as otherwise the precipitate would consist of a basic salt, which would not become black when boiled.

335. (C) *Ferrocyanide of potassium* ($K_2Fcy+3Aq$) gives, even in very dilute solutions, a mahogany colored precipitate of ferrocyanide of copper ($\mathrm{Cu_2Fcy}$), which is insoluble in dilute acids.

336. (C) A piece of clean *iron* (a knife blade), when placed in a solution containing copper, causes a precipitation of metallic copper on its surface.

$$CuO,SO_3+\mathrm{Fe}=\mathrm{Cu}+FeO,SO_3.$$

This is an extremely delicate test, and by this means the whole of the copper may be removed from a liquid, especially if a slight excess of acid is present.

337. *Identification of metallic copper.*—Dissolve a very small piece of copper in dilute nitric acid; test one part of the solution with ammonia (333), and the other with ferrocyanide of potassium (335).

[For the blowpipe test, see (120).]

SECTION VI.

Silver (Ag).

A solution of nitrate of silver (AgO,NO_5) may be used.

338. (C) Most of the salts of silver when exposed to light, especially when in contact with organic matter, gradually become more or less purple, and eventually nearly black, owing to partial decomposition.

339. *Hydrosulphuric acid* (HS) and *hydrosulphate of ammonia* (NH_4S,HS) throw down a black precipitate of sulphide of silver (AgS), which is insoluble in cold dilute acids, but soluble in boiling nitric acid.

340. (C) *Hydrochloric acid* (HCl) or a solution of *chloride of sodium* ($NaCl$) produces in solutions of silver a white curdy precipitate of chloride of silver ($AgCl$), which is insoluble in water and in nitric acid, but readily soluble in ammonia, and very sparingly so in an excess either of hydrochloric acid or chloride of sodium.

$$AgO,NO_5+NaCl=AgCl+NaO,NO_5.$$

If the ammoniacal solution be neutralized with nitric acid, the chloride is reprecipitated.

341. (C) *Phosphate of soda* ($2NaO,HO,PO_5+24Aq$) throws down a pale yellow precipitate of tribasic phosphate of silver ($3AgO,PO_5$), which is soluble both in nitric acid and ammonia.

342. *Identification of metallic silver.*—Dissolve a scrap of silver in dilute nitric acid, with the aid of heat, and ascertain the presence of the metal by adding hydrochloric acid, and dissolving the precipitate in ammonia.

[For the blowpipe test, see (117).]

SECTION VII.

Tin (Sn).

A. *As protoxide* (SnO) *or some corresponding compound.*

A solution of protochloride of tin (*SnCl*) may be used.

343. (C) *Hydrosulphuric acid* (HS) gives in solutions of the protosalts of tin, either neutral or with excess of acid, a dark brown precipitate of protosulphide of tin (SnS), which is soluble in hydrosulphate of ammonia, if it contains an excess of sulphur. The solution is facilitated by heating.

344. (C) *Hydrosulphate of ammonia* (NH_4S,HS) also throws down the brown protosulphide, which is soluble in excess, provided a little free sulphur is present in it, which is always the case when the hydrosulphate has a yellow color. If the solution thus formed be neutralized with hydrochloric acid, a yellow precipitate of the persulphide (SnS_2) is produced, which was formed by the action of the excess of sulphur in the hydrosulphate upon the protosulphide. $SnS+S=SnS_2$.

345. (C) *Chloride of mercury* (HgCl) produces, even in very dilute solutions, at first a white precipitate of subchloride (Hg_2Cl), which is often highly crystalline, and gives a silky appearance to the liquid. If a sufficient quantity of the tin salt be present, this white precipitate is converted after a time into gray metallic mercury.

B. *As peroxide of tin* (SnO_2)[1] *or some corresponding compound.*

A solution of the perchloride ($SnCl_2$) may be used.

346. (C) *Hydrosulphuric acid* (HS) gives, in acid solutions, a pale yellow precipitate of bisulphide of tin (SnS_2), which is in some cases formed only on heating.

347. (C) *Hydrosulphate of ammonia* (NH_4S,HS) also

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[1] **Also called *stannic acid*.**

throws down the yellow persulphide, which is readily soluble in excess.

348. *Ammonia* (NH_3) and *potash* (KO) throw down, from acid solutions, a bulky white precipitate of hydrated peroxide of tin (SnO_2,HO), which is soluble in an excess of the precipitant, especially when potash is used and heat applied, forming a compound called stannate of potash (KO,SnO_2) in which the peroxide of tin plays the part of an acid.

349. The hydrated peroxide, when thus formed by precipitation with potash, is readily soluble both in potash and nitric acid, in which respect it differs from that formed by the action of nitric acid on metallic tin, though both yield the same results when analyzed.

350. *Sesquicarbonate of ammonia*, in acid solutions, produces a white precipitate of hydrated peroxide of tin, which is not dissolved on boiling with an excess of the carbonate.

351. *Identification of metallic tin.*—Boil a small fragment of tin with dilute nitric acid; it is converted into a white insoluble powder, which is metastannic acid (Sn_5O_{10}). Boil another portion of the metal with hydrochloric acid, which slowly dissolves it as protochloride of tin ($SnCl$); pour a little of the solution into another tube and test with chloride of mercury (345). Continue to boil the rest of the tin with the acid, and add dilute nitric acid very carefully at intervals, until the whole is dissolved. Test the bichloride of tin thus produced with hydrosulphuric acid (346), and with carbonate of ammonia (350).

[For the blowpipe test, see (118).]

SECTION VIII.

Bismuth (Bi).

A solution of the chloride ($BiCl_3$) may be used.

352. (C) Mix a concentrated solution of the chloride with a considerable quantity of *water*, which causes a white precipitate of oxychloride of bismuth ($BiCl_3,2BiO_3$).

A similar decomposition takes place when solutions of many of the soluble salts of bismuth are diluted with much water. The precipitates thus formed are usually distinguishable from those produced under the same circumstances in solutions of antimony, by being insoluble in tartaric acid.

353. *Hydrosulphuric acid* (HS) and *hydrosulphate of ammonia* (NH_4S,HS) throw down, from solutions of bismuth, a black precipitate of sulphide of bismuth (BiS_3), which is insoluble in cold dilute acids, but soluble in hot nitric acid.

354. *Ammonia* (NH_3) gives a white precipitate of hydrated oxide (BiO_3,HO), which is insoluble in an excess of the precipitant. If the precipitate be redissolved in as little hydrochloric acid as possible, the solution will become milky when mixed with a large quantity of water (352).

[For the blowpipe test, see (116).]

Summary of Class IV.

355. In order to ascertain if a solution contains either of the metals belonging to this class, add hydrochloric acid until it is distinctly acid, and if any precipitate be produced,[1] continue to add hydrochloric acid and heat gently, in order to be quite sure that the precipitate does not redissolve[2] (304), in which case it is probably either subchloride of mercury, chloride of silver, or chloride of lead. To determine between these, allow the precipitate to settle in the tube, pour off the liquid, and boil the precipitate with water; chloride of lead will be dissolved, and the presence of lead may be confirmed by adding dilute sulphuric acid (327); if the precipitate remains undissolved, let it subside again, pour off the water, and shake the precipitate with ammonia; if it dissolves, it is chloride of silver, and may be again precipitated by nitric acid; if it is blackened, it is subchloride of mercury, and indicates the presence of mercury in the solution in the form of suboxide (Hg_2O).

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[1] If no precipitate be produced, pass on to (356).

[2] If the precipitate redissolves, pass on to (356).

356. If no precipitate be produced by hydrochloric acid, or if the precipitate be afterwards redissolved, add to the same solution hydrosulphuric acid (which, if employed in solution, must be thoroughly saturated), as long as it produces any change (319); if any precipitate separates, its color and appearance must be carefully observed; if it be *white*, and uniformly diffused through the liquid so that separate flakes cannot be perceived, it is only sulphur, and may be disregarded;[1] if it be distinctly *orange-red*, it is sulphide of antimony, and the presence of that metal should be confirmed by additional tests (305, 307, 308); if the precipitate be *pale yellow*, it is probably bisulphide of tin, and indicates the presence of tin in the form of binoxide (SnO_2), or some corresponding compound; this result should be confirmed by (347) and (350). If the precipitate be *bright yellow*, it is probably tersulphide of arsenic, indicating the presence of arsenious acid (AsO_3) or of some corresponding compound; confirm by (274) and (275). A decidedly *brown*[2] precipitate is probably protosulphide of tin, indicating the presence of tin as protoxide (SnO) or some corresponding compound; confirm by (344) and (345). A *black* precipitate may be either sulphide of lead, bisulphide of mercury (319), sulphide of bismuth, or sulphide of copper. To distinguish between these, separate portions of the original solution must be tested according to the directions given in (327), (329), (321), (324), (352), and (333).

357. Should no precipitate, except sulphur, be produced by hydrosulphuric acid, the solution must be boiled; if a *yellow* precipitate be then produced, it is probably either bisulphide of tin, which may be further proved by (347) and (350); or tersulphide of arsenic resulting from the decomposition of arsenic acid (299); the precipitate must be tested by (274) and (275).

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[1] If no precipitate (except sulphur) be produced, pass on to (357).

[2] It is advisable not to rely much upon the difference between brown and black in this precipitate.

CHAPTER VI.

NON-METALLIC BODIES.

SECTION I.

Nitrogen.

358. The most frequent forms of combination in which this element is met with are ammonia (NH_3) and its salts, nitric acid (HO,NO_5) and the nitrates, cyanogen (C_2N) and its compounds, and nitrogenized organic substances which contain, beside nitrogen, carbon, hydrogen, and sometimes oxygen and sulphur. A method of recognizing nitrogen in this last form has been given at (111), and the tests for ammonia have been already described in the section on ammonium. The recognition of cyanogen will be treated of hereafter (429). It remains to be seen how nitrogen is recognized when in the form of nitric acid or a nitrate.

Nitric Acid (HO,NO_5).

359. Heat a little powdered nitrate of potash (KO,NO_5), or some other nitrate, with *concentrated sulphuric acid* (HO,SO_3); vapors of nitric acid (HO,NO_5) are evolved, mingled usually with red-brown vapors of peroxide of nitrogen (NO_4).

360. (C) Mix some solution of nitrate of potash with about an equal volume of *concentrated sulphuric acid*, add a few *copper filings*, and apply heat; red-brown vapors are produced, which are seen to the greatest advantage by looking down the tube. In this experiment, the nitric acid, which is set free by the sulphuric acid, is deoxidized by the copper, being converted into nitric oxide (NO_2), which absorbs oxygen when it issues into the air, forming red fumes of nitrous acid (NO_3) and peroxide of nitrogen (NO_4).

$$KO,NO_5 + Cu_3 + 4(HO,SO_3) = 3(CuO,SO_3) + KO,SO_3 + 4HO + NO_2.$$

361. (C) Shake a small crystal of *protosulphate of iron* (FeO,SO_3) with solution of nitrate of potash for a few seconds without waiting till it is entirely dissolved, and pour *concentrated sulphuric acid* very carefully down the side of the tube (slightly inclined), so that it may form a distinct layer at the bottom; a brown or pink band will appear where the two layers of liquid are in contact. If the quantity of nitric acid present be very minute, several minutes may elapse before the reaction is perceptible. The brown compound is formed by the union of protosulphate of iron with nitric oxide (NO_2), the other three equivalents of oxygen of the nitric acid having combined with another portion of the protoxide of iron to convert it into sesquioxide, which combines with the sulphuric acid to form the sesquisulphate ($Fe_2O_3,3SO_3$).

$$10(FeO,SO_3)+4(HO,SO_3)+KO,NO_5=3(Fe_2O_3,3SO_3)+ \\ KO,SO_3+NO_2,4(FeO,SO_3)+4HO.$$

If the tube is shaken or heated, the brown compound is decomposed, and the color disappears.

[For the blowpipe tests, see (107) and (112.)]

SECTION II.

Sulphur.

362. Sulphur is most frequently met with in the form of sulphuric acid (HO,SO_3) or a sulphate; much less frequently as sulphurous acid (SO_2) or a sulphite, or as a hyposulphite (a salt of hyposulphurous acid, S_2O_2). It also commonly occurs as hydrosulphuric acid or sulphuretted hydrogen (HS), and as a sulphide (a combination of sulphur with a metal).

Sulphuric acid (HO,SO_3).

363. Mix a few drops of strong sulphuric acid (oil of vitriol) with about an equal quantity of water, in a test-tube, and observe the heat evolved. Dilute the mixture with three or four volumes of water, place a drop upon a piece of filter paper, and dry it without scorching the paper; observe the carbonization of that part of the paper which was moistened with the acid.

364. (C) Test a solution of sulphate of soda (NaO,SO_3),

or some other sulphate, with *chloride of barium ;* a heavy white precipitate of sulphate of baryta, is obtained, which is insoluble in dilute hydrochloric acid.

[For the blowpipe test, see (122).]

Sulphurous Acid (SO_2).

365. (C) Heat a little sulphite of soda (NaO,SO_2) with *dilute hydrochloric acid;* sulphurous acid will be disengaged, and may be recognized by its odor of burning sulphur.

366. (C) Acidulate a solution of sulphite of soda strongly with *hydrochloric acid,* and introduce a fragment of *zinc:* sulphuretted hydrogen (HS) will be evolved, and may be known by its odor, and by its action upon paper moistened with acetate of lead (372).

367. *Chloride of barium* and *chloride of calcium* produce in the solution of a sulphite (*e.g.*, sulphite of soda) white precipitates, which are soluble in dilute hydrochloric acid.

368. *Nitrate of silver* produces a white precipitate of sulphite of silver (AgO,SO_2) which becomes reduced to the metallic state when heated in the liquid.

$$AgO,SO_2 + HO = Ag + HO,SO_3.$$

Hyposulphurous Acid (S_2O_2).

369. (C) Add a little *hydrochloric acid* to solution of hyposulphite of soda (NaO,S_2O_2); after a short time the solution becomes milky, from separation of sulphur, and evolves the odor of sulphurous acid. The change is accelerated by heat.

370. Prepare a little chloride of silver by precipitating nitrate of silver with chloride of sodium; wash it by decantation, and shake it with solution of hyposulphite of soda, which will readily dissolve it, producing a very sweet solution.

Hydrosulphuric Acid (*HS*) *and Metallic Sulphides.*

371. (C) *Acetate of lead* ($PbO,C_4H_3O_3$) gives, with solution of hydrosulphuric acid or a sulphide, a black precipitate of sulphide of lead (PbS).

372. (C) Pour a little *dilute hydrochloric acid* upon a small fragment of sulphide of iron (FeS) in a test-tube; hydrosulphuric acid is evolved, which may be known by its odor, and if a piece of filter paper, moistened with solution of acetate of lead, be held at the mouth of the tube, it will be coated with a black shining deposit of sulphide of lead (PbS).

373. Add a little *dilute hydrochloric acid* to a drop or two of yellow hydrosulphate of ammonia, hydrosulphuric acid is given off, and the liquid becomes milky from the separation of sulphur.

When hydrosulphate of ammonia (NH_4S,HS) is first prepared, it is colorless, but a portion of the hydrosulphuric acid is gradually decomposed by the atmospheric oxygen, which combines with its hydrogen to form water, leaving an equivalent of free sulphur which dissolves in the hydrosulphate, giving it a yellow color, and is precipitated on adding an acid.

$$(NH_4S+S)+HCl=NH_4Cl+HS+S.$$

374. Boil a little sulphide of iron with *concentrated nitric acid;* the metal is oxidized and dissolved, whilst the sulphur separates in gray flakes which rise to the surface of the liquid, and eventually fuse into yellow globules. Dilute the solution with water, filter it, and test with chloride of barium for the sulphuric acid which has been formed by the oxidation of a portion of the sulphur.

[For the blowpipe test, see (119).]

SECTION III.

Phosphorus.

375. This element is generally met with in the form of tribasic phosphoric acid ($3HO,PO_5$) or a tribasic phosphate.

Tribasic Phosphoric Acid ($3HO,PO_5$).

A solution of common phosphate of soda ($2NaO,HO,PO_5+24Aq$) may be used.

376. (C) Mix a little solution of *sulphate of magnesia* with *chloride of ammonium* and *ammonia* (209), and add

the mixture to a solution of phosphate of soda; a white crystalline precipitate of phosphate of magnesia and ammonia ($2MgO,NH_4O,PO_5$) will be produced. If the solution be dilute, the precipitate is formed only after standing; its deposition is promoted by rubbing the side of the tube with a glass rod. (Arsenic acid gives a similar reaction.)

377. (C) *Perchloride of iron* (Fe_2Cl_3) gives a nearly white precipitate of phosphate of iron (Fe_2O_3,PO_5) which is insoluble in acetic acid, but dissolves readily in hydrochloric acid. (Arsenic acid gives a similar reaction.)

378. (C) Dissolve a very little bone-ash (phosphate of lime, $3CaO,PO_5$) in as little dilute hydrochloric acid as possible, add a little perchloride of iron, and some acetate of potash or acetate of ammonia (prepared by mixing ammonia with acetic acid to acid reaction); collect the precipitated phosphate of iron upon a filter, wash it, dissolve off the filter with a little warm dilute hydrochloric acid, mix the solution with excess of ammonia and hydrosulphate of ammonia, boil, filter off the black sulphide of iron, and test the solution for phosphoric acid as in (376).[1]

379. *Chloride of barium* produces in a solution of phosphate of soda a white precipitate of phosphate of baryta ($2BaO,HO,PO_5$), which is soluble in dilute hydrochloric acid.

380. *Chloride of calcium* gives a white precipitate of phosphate of lime ($2CaO,HO,PO_5$), which readily dissolves in acetic acid; the solution gives a white precipitate with perchloride of iron.

381. (C) *Nitrate of silver* gives a yellow precipitate of tribasic phosphate of silver ($3AgO,PO_5$), which is soluble both in ammonia and nitric acid (280). (Arsenic acid gives a brown precipitate (298).)

$$3(AgO,NO_5)+2NaO,HO,PO_5=3AgO,PO_5+2(NaO,NO_5)\\+HO,NO_5.$$

382. Heat a crystal of common phosphate of soda to redness on platinum foil or wire; dissolve it in water, and

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[1] Molybdate of ammonia (NH_4O,MoO_3) produces in solutions of the earthy phosphates in nitric acid a crystalline yellow precipitate, consisting of molybdic acid, phosphoric acid, and ammonia.

test with nitrate of silver. Instead of the yellow tribasic phosphate ($3AgO,PO_5$), it will give a white granular precipitate of the bibasic phosphate ($2AgO,PO_5$), corresponding to the bibasic phosphate of soda ($2NaO,PO_5$), into which the common phosphate has been converted by the expulsion of the basic water.

SECTION IV.

Chlorine.

383. The most frequently occurring forms of combination of chlorine are hydrochloric acid (HCl), and the chlorides formed by the union of chlorine with a metal. It is also occasionally met with in combination with oxygen, as chloric acid (ClO_5) and the chlorates, or hypochlorous acid (ClO) and the hypochlorites.

Hydrochloric acid (HCl), and metallic chlorides.

384. (C) Add *nitrate of silver* to a solution of chloride of sodium, or any other chloride ; a white curdy precipitate of chloride of silver ($AgCl$) will separate, unless the solution be extremely dilute, when a milkiness only would be perceived.

$$AgO,NO_5+NaCl=AgCl+NaO,NO_5.$$

Pour a part of the liquid containing the precipitate into another tube, and test the remainder with nitric acid, which will not dissolve it, although it may cause it to shrink together and undergo an apparent diminution.

Shake the other portion with ammonia, which will immediately dissolve it; on adding nitric acid to the ammoniacal solution, the chloride of silver is again precipitated.

385. (C) Pour a little *concentrated sulphuric acid* upon solid chloride of sodium; it froths and evolves hydrochloric acid, which may be known by its odor, and by the gray fumes which it produces in moist air.

386. (C) Heat a little solid chloride of sodium, or other chloride to be tested, with *dilute sulphuric acid* and *binoxide of manganese;* chlorine is evolved, and may be recognized by its odor, and by its property of bleaching moistened test-papers.

Chloric Acid (HO,ClO_5).

387. (C) Place a small fragment of chlorate of potash (KO,ClO_5) in a test-tube, and pour upon it a few drops of strong sulphuric acid; the chlorate is decomposed, yielding perchlorate of potash (KO,ClO_7) and peroxide of chlorine (ClO_4), which gives the mixture a red or yellow color, and escapes in the form of a greenish yellow gas of very peculiar odor.

$$3(KO,ClO_5)+2(HO,SO_3)=2(KO,SO_3)+KO,ClO_7+2ClO_4+2HO.$$

On applying a gentle heat to the mixture, sharp detonations are often perceived, arising from the decomposition of the peroxide of chlorine into its elements. If any chloride be present in the chlorate, the detonations often take place in the cold, in consequence of the mutual decomposition between the hydrochloric and chloric acids which are then evolved.

388. (C) Heat a small fragment of chlorate of potash on charcoal, before the blowpipe; a deflagration will ensue, which is more violent than with nitrate of potash.

389. (C) Heat a little of the chlorate in a small sealed tube; it is decomposed, and if the heat be continued long enough, the whole of the oxygen is given off, chloride of potassium (KCl) being left. $KO,ClO_5=KCl+O_6$. The disengagement of oxygen may be proved by introducing into the tube a match with a spark at the end.

Dissolve the residue out of the tube with water, and test the solution with nitrate of silver, which will give a precipitate of chloride of silver, insoluble in nitric acid. The formation of a chloride under the action of heat is the best proof that the acid is chloric and not nitric. Chlorate of potash, if pure, gives no precipitate with nitrate of silver.

Hypochlorous Acid (ClO).

Solution of chloride of lime ($CaO,ClO+CaCl$) may be used.

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390. (C) Solutions of the hypochlorites bleach indigo,

litmus, and many other organic coloring matters very readily, when mixed with a little dilute sulphuric acid, which liberates chlorine from them, recognized by its odor.

$(CaO,ClO + CaCl) + 2(HO,SO_3) = 2(CaO,SO_3) + 2HO + Cl_2$.

391. (C) Sulphate of manganese (MnO,SO_3) gives a black precipitate of binoxide of manganese.

SECTION V.

Iodine.

392. We are most likely to meet with iodine, either in the free state or in combination with hydrogen, as hydriodic acid, or with a metal as an iodide.

Free iodine would be at once recognized by its resembling black lead in appearance, and furnishing violet vapors of peculiar odor when heated.

Hydriodic acid (HI) and metallic iodides.

A solution of iodide of potassium may be used.

393. (C) *Starch* ($C_{12}H_{10}O_{10}$) added to a solution of an iodide, gives no reaction unless the iodine is set free by the addition of some oxidizing agent. This may be effected by *concentrated nitric acid*, which should be added drop by drop, when the purple or blue color of the iodized starch will be perceived. Strictly speaking, the action is not due to the nitric acid, but to the nitrous acid (NO_3), and peroxide of nitrogen (NO_4), which are contained in the ordinary specimens of the acid.

$$KI + NO_3 = KO + NO_2 + I.$$

394. (C) *Concentrated sulphuric acid*, heated with an iodide, liberates iodine, recognized by its violet-colored vapor, which imparts a blue color to paper impregnated with starch.[1]

$$KI + 2(HO,SO_3) = KO,SO_3 + SO_2 + 2HO + I.$$

395. (C) *Nitrate of silver* gives a pale yellow precipi-

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[1] **Some hydrosulphuric acid is evolved at the same time.**

tate of iodide of silver (AgI), insoluble in nitric acid and in ammonia (384).

396. (C) *Perchloride of mercury* (HgCl) gives a brilliant red precipitate of iodide of mercury (HgI), soluble in an excess either of the perchloride or of iodide of potassium.

397. *Bichloride of platinum* produces a dark brownish-red color.[1]

[For the blowpipe test, see (108).]

SECTION VI.

Fluorine.

398. This element is likely to be met with either as hydrofluoric acid (HF), or as the fluoride of a metal.

Hydrofluoric acid (HF) and metallic fluorides.

399. (C) Place a little powdered fluor spar (fluoride of calcium, CaF), or other fluoride to be tested, in a platinum crucible or leaden cup, moisten it with *concentrated sulphuric acid*, and cover the crucible with a piece of window glass; after allowing it to remain for some time upon a warm sand-bath, the glass will be found corroded by the vapor of hydrofluoric acid.

$$CaF + HO,SO_3 = CaO,SO_3 + HF.$$

400. (C) This test may also be applied by moistening a little fluor spar upon a piece of glass with strong sulphuric acid, heating gently, and washing off the mass after a few minutes, when the glass beneath will be found corroded.

401. (C) Mix some fluor spar with *sand* (SiO_2), and heat the mixture with *concentrated sulphuric acid* in a dry test-tube furnished with a perforated cork, carrying a piece of glass tube moistened in the inside; fluoride of silicon (SiF_2) will be evolved, and will be decomposed by the water, depositing a white coating of silica upon the sides of the moistened tube:

$$2CaF + SiO_2 + 2(HO,SO_3) = SiF_2 + 2(CaO,SO_3) + 2HO;$$
$$\text{and } 3SiF_2 + 2HO = SiO_2 + 2(HF,SiF_2).$$

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[1] Bichloride of palladium is often a useful test for iodides, with which it produces a black precipitate.

402. Fuse upon platinum foil a mixture of very finely powdered fluor spar with three or four parts of carbonate of soda; boil the fused mass upon the foil in a dish with water, to dissolve out the fluoride of sodium.

$$CaF+NaO,CO_2=NaF+CaO,CO_2.$$

Filter the solution, and neutralize it carefully with acetic acid.

403. *Chloride of barium* added to a portion of this solution gives a translucent precipitate of fluoride of barium (BaF), soluble in dilute hydrochloric acid.

404. *Chloride of calcium* gives a similar precipitate, almost insoluble in acetic acid.

[For the blowpipe test, see (109).]

SECTION VII.

Boron.

405. The ordinary forms of combination in which boron is met with are boracic acid (BO_3), and the borates.

Boracic acid (BO_3).

406. (C) Add *dilute hydrochloric acid*, drop by drop, to a solution of borax ($NaO,2BO_3$), until it distinctly reddens blue litmus paper; dip one end of a slip of *turmeric paper* into the acid solution, and dry it at a little distance above the flame, being careful not to scorch it; the yellow color of the turmeric will be changed to a bright orange-red.[1]

407. (C) Mix a solution of borax, or other borate to be tested, in a small dish, with *strong sulphuric acid* and alcohol, kindle the mixture, by means of a glass rod dipped into it, and stir it well, in a dark place; the flame will be tinged with green, especially at the edge, a part of the boracic acid being deoxidized, and the boron burning with a green flame.[2]

408. Make a very strong solution of borax, by boiling

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[1] A solution containing 0.25 per cent. of boracic acid will respond distinctly to this test.

[2] If copper be present it may also impart a green flame, and must be separated before applying this test.

with water, allow it to cool, and add *strong hydrochloric acid;* feathery crystals of boracic acid (HO,BO_3+2Aq) will be deposited.

409. *Chloride of barium*, added to a solution of borax, precipitates biborate of baryta ($BaO,2BO_3$) which is readily soluble in dilute hydrochloric acid.

$$BaCl+NaO,2BO_3=NaCl+BaO,2BO_3.$$

410. *Chloride of calcium* gives a white precipitate of biborate of lime ($CaO,2BO_3$) readily soluble in acetic acid.

Both this precipitate and the preceding one are soluble in chloride of ammonium, so that when dissolved in hydrochloric acid, they are not generally precipitated by ammonia.

411. *Nitrate of silver* (AgO,NO_5) gives a white precipitate of borate of silver (AgO,BO_3) which is easily soluble in nitric acid.

[For the blowpipe test, see (139).]

SECTION VIII.

Silicon.

412. Silicic acid (SiO_2) and the silicates are the forms in which this element is generally met with.

Silicic acid (SiO_2).

413. (C) Mix a little finely-powdered sand (insoluble variety of silica) with four parts of dry *carbonate of soda*, and fuse the mixture for two or three minutes upon platinum foil, directing the blowpipe flame upon the under side; effervescence takes place, from the carbonic acid, and silicate of soda is formed.

$$NaO,CO_2+SiO_2=NaO,SiO_2+CO_2.$$

Soak the platinum foil in water (in a dish), and the mass will dissolve; acidify the solution with hydrochloric acid, and divide it into two parts.

414. (C) Evaporate one part (acidulated with hydrochloric acid) to dryness (in a dish), and treat the residue with *dilute hydrochloric acid;* the chloride of sodium will

be dissolved, but white flakes of insoluble silica will be left.

415. To the other part add a slight excess of ammonia; soluble silica will separate as a gelatinous precipitate, which might easily be mistaken for alumina (216).

[For the blowpipe tests, see (138) and (138*a*).]

SECTION IX.

Carbon.

416. The forms of combination in which carbon is commonly met with in the analysis of inorganic substances are carbonic acid (CO_2) and the carbonates; but since this element is present in all organic (*i.e.*, animal and vegetable) substances, the various forms of combination in which it may present itself are far too numerous to be considered in this work. There are, however, a few other acids containing carbon which are liable to be encountered in the ordinary course of analysis, and are here enumerated, their chief source, whether natural or artificial, being at the same time recalled to the mind of the student.

Oxalic acid (HO,C_2O_3+2Aq); wood sorrel; rhubarb; action of nitric acid on starch or sugar.

Acetic acid ($HO,C_4H_3O_3$); vinegar.

Hydrocyanic acid (H,C_2N); bitter almonds; prussiate of potash.

Hydroferrocyanic acid (H_2,C_6N_3Fe); yellow prussiate of potash.

Tartaric acid ($2HO,C_8H_4O_{10}$); juice of the grape.

Citric acid ($3HO,C_{12}H_5O_{11}+2Aq$); lemon-juice.

Tannic acid ($C_{54}H_{22}O_{34}$); gall nuts.

Gallic acid ($3HO,C_{14}H_3O_7+2Aq$); gall nuts.

Benzoic acid ($HO,C_{14}H_5O_3$); gum benzoin.

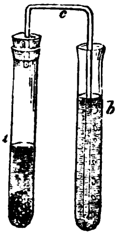
Carbonic Acid (CO_2).

417. (C) Add *dilute hydrochloric acid* to solution of carbonate of soda; notice the effervescence, and the faint odor of the carbonic acid evolved. Put a little powdered marble (CaO,CO_2) or chalk into a test-tube, pour upon it some dilute hydrochloric acid, and incline the mouth of

the tube over that of another, half filled with lime-water, so as to pour the carbonic acid gas into the second tube, but not any of the liquid; close the tube containing lime-water with the thumb, and shake it, when it will become milky, from the separation of carbonate of lime.

If the quantity of carbonic acid be very small, it is better to pass it into lime-water, as represented in Fig. 71; *a* representing the test-tube containing the carbonate from which the gas is conducted by the bent tube *c* into the lime-water in *b;* a very gentle heat may be applied to promote the disengagement of the carbonic acid.[1]

Fig. 71.



418. *Chloride of barium* or *Chloride of calcium* added to a solution of a carbonate, will produce a white precipitate of carbonate of baryta (BaO,CO_2) or of lime (CaO,CO_2) readily soluble, with effervescence, in acetic acid.

Oxalic Acid (HO,C_2O_3).

419. (C) Add some *chloride of calcium* ($CaCl$) to a solution of an oxalate (*e.g.*, oxalate of ammonia); a white precipitate of oxalate of lime (CaO,C_2O_3+2Aq) is produced, even though the solution be very dilute.

$$CaCl+NH_4O,C_2O_3=CaO,C_2O_3+NH_4Cl.$$

The precipitate will be found to be insoluble in acetic acid, but readily soluble in hydrochloric acid.

420. *Chloride of barium*, added to oxalate of ammonia, will give a white precipitate of oxalate of baryta (BaO,C_2O_3), which is soluble in dilute hydrochloric acid.

421. Heat a little crystallized oxalic acid (HO,C_2O_3+2Aq) in a small tube; observe that a great part volatilizes unchanged, condensing in fine needles on the cooler part of the tube.

422. (C) Pour a little *strong sulphuric acid* upon some oxalic acid or any oxalate to be tested, and apply heat;

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[1] Some natural carbonates, especially dolomite (carbonate of lime and magnesia), and native carbonate of iron, give scarcely any effervescence with hydrochloric acid in the cold.

effervescence ensues, from the disengagement of carbonic acid and carbonic oxide.

$$HO,C_2O_3=HO+CO+CO_2.$$

The carbonic oxide may be recognized by its burning with a blue flame when the mouth of the tube is applied to a light.

423. Heat a little oxalate of lime (CaO,C_2O_3) with dilute hydrochloric acid, which will dissolve it without effervescence; neutralize the acid with ammonia, and the oxalate will be precipitated.

424. Place some oxalate of lime upon a platinum foil, and heat it to low redness; the salt is scarcely changed in appearance, but if it be now moistened with hydrochloric acid, it will effervesce violently, having been converted into carbonate of lime by calcination.

$$CaO,C_2O_3=CaO,CO_2+CO.$$

If the solution in hydrochloric acid be boiled, to expel free carbonic acid, and neutralized with ammonia, no precipitate will occur if the oxalate has been completely decomposed.

Acetic Acid ($HO,C_4H_3O_3$).

425. (C) Heat a little acetate of lead ($PbO,C_4H_3O_3$) in a small tube, and observe the peculiar odor of acetone ($C_6H_6O_2$) which it emits.

$$2(PbO,C_4H_3O_3)=C_6H_6O_2+2PbO+2CO_2.$$

426. (C) Pour a little *strong sulphuric acid* upon some acetate of lead, or other acetate to be tested in a test-tube, and apply heat, when acetic acid will be evolved; which may be recognized by its odor.

427. (C) Repeat the last experiment with the addition of a little *alcohol* ($C_4H_6O_2$); the peculiar agreeable odor of acetic ether ($C_4H_5O,C_4H_3O_3$) will be perceived, especially when the mixture has cooled.

$$PbO,C_4H_3O_3+C_4H_6O_2+HO,SO_3=C_4H_5O,C_4H_3O_3 \\ +2HO+PbO,SO_3.$$

428. Add ammonia, drop by drop, to acetic acid, till the liquid is slightly alkaline, evaporate it gently in a

dish till the alkaline reaction is no longer perceptible, and add *perchloride of iron*; a red color (but no precipitate) is produced, due to the formation of peracetate of iron ($Fe_2O_3,3C_4H_3O_3$); the color does not disappear on adding perchloride of mercury.

Hydrocyanic Acid (HC_2N), *or the cyanide of a metal.*

429. (C) Mix a little hydrocyanic acid, or other solution to be tested for cyanogen, with solution of *potash*, add a little solution of *protosulphate of iron* (FeO,SO_3), then some *perchloride of iron*, and finally an excess of *dilute hydrochloric acid;* a precipitate of Prussian blue (Fe_4Fcy_3) is obtained, or if there be very little hydrocyanic acid present, a blue color is apparent in the solution.[1]

430. (C) To a little hydrocyanic acid, add a few drops of yellow *hydrosulphate of ammonia*, and evaporate the solution nearly to dryness in a small porcelain dish (until all odor of the hydrosulphate has disappeared); sulphocyanide of ammonium (NH_4CyS_2) is formed, and on adding a little perchloride of iron, will give the blood-red color of sulphocyanide of iron ($Fe_2(CyS_2)_3$), which is bleached by perchloride of mercury.

$$HCy + NH_4S + S_2 = NH_4CyS_2 + HS.$$

431. *Nitrate of silver* (AgO,NO_5) added to hydrocyanic acid causes a white precipitate of cyanide of silver ($AgCy$), which is sparingly soluble in cold dilute nitric acid, but dissolves in the concentrated acid, especially on heating. Cyanide of silver also dissolves readily in ammonia, and is partly precipitated again by the careful addition of excess of nitric acid.

When cyanide of silver is dried, and heated to redness, it evolves cyanogen; if the experiment be performed in a small glass tube closed at one end, the cyanogen may be recognized by its odor, and by its burning with

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[1] The color varies through different shades of blue and green according to the proportions in which the iron salts are added, but either color may be taken, if well marked, as affording evidence of the presence of hydrocyanic acid. The change by which the Prussian blue is produced is explained in the following equations:—

$$HCy + KO = KCy + HO,$$
$$3KCy + FeO,SO_3 = K_2Cy_3Fe\ (=K_2Fcy) + KO,SO_3, \text{ and}$$
$$3K_2Fcy + 2Fe_2Cl_3 = 6KCl + Fe_4Fcy_3.$$

a pink flame. If the cyanide of silver be heated in a crucible, only metallic silver remains, which is entirely dissolved by nitric acid.

432. *Dilute sulphuric* or *hydrochloric acid* decomposes the cyanides (*e.g.*, cyanide of potassium), evolving hydrocyanic acid, which may be recognized by its peculiar odor.

Hydroferrocyanic Acid ($H_2Cy_3Fe=H_2Fcy$), *or the ferrocyanide of a metal.*

433. (C) *Perchloride of iron* (Fe_2Cl_3), added to an acid or neutral solution of a ferrocyanide (*e.g.*, ferrocyanide of potassium), causes a dark blue precipitate of Prussian blue (Fe_4Fcy_3).

434. Nitrate of silver gives a white precipitate of ferrocyanide of silver (Ag_2Fcy), not dissolved by dilute nitric acid or ammonia; decomposed by heat, the silver being reduced to the metallic state.

Tartaric Acid ($2HO,C_8H_4O_{10}$).

435. (C) Heat a small crystal of the acid on platinum foil; it at first fuses, and is afterwards decomposed, with deposition of carbon, and consequent blackening. A peculiar and characteristic odor is at the same time emitted.

436. Heat a small fragment of bitartrate of potash ($KO,HO,C_8H_4O_{10}$) on platinum foil, before the blowpipe or over a spirit lamp. The tartaric acid is thus decomposed, and carbonate of potash (KO,CO_2) is at the same time formed. Place the fragment in a test-tube, and add a few drops of dilute hydrochloric acid, when it will effervesce, showing the presence of carbonic acid.

437. (C) *Chloride of calcium* (*CaCl*) throws down in neutral solutions containing tartaric acid[1] a white precipitate of tartrate of lime ($2CaO,C_8H_4O_{10}$), which is soluble, after washing, in a cold solution of potash; if the potash

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[1] To prepare a neutral solution of an organic acid for testing, the acid should be dissolved in water, and carbonate of soda carefully added till the liquid is slightly alkaline, a few drops of the acid solution are added till a slight acid reaction is perceptible, the liquid is heated to expel free carbonic acid, and is rendered slightly alkaline by addition of ammonia, the excess of the latter being afterwards expelled by heating the solution until it is perfectly neutral.

solution be heated, however, the tartrate of lime separates as a bulky precipitate, but redissolves as the solution cools. This precipitate is soluble in chloride of ammonium; the presence of ammoniacal salts in the liquid will therefore prevent its formation. Acetic acid also dissolves the tartrate of lime.

438. *Carbonate of potash*, added to tartaric acid, in quantity insufficient to neutralize it, produces a granular crystalline precipitate of bitartrate of potash ($KO,HO,C_8H_4O_{10}$), the separation of which is much promoted by brisk stirring (188).

439. Tartaric acid and the tartrates, when present in solutions of the persalts of iron, prevent the precipitation of the hydrated peroxide ($Fe_2O_3,3HO$) when ammonia or potash is added. This is owing to the formation of double tartrates of iron and the alkali, which are soluble in water, and are not decomposed by an excess of alkali. Tartaric acid also prevents the precipitation of alumina, protoxide of manganese, and some other oxides, under similar circumstances.

439*a*. When tartaric acid is heated with *strong sulphuric acid*, it blackens at once; if the mouth of the tube be applied to a light, it will be found that carbonic oxide is passing off in small quantity.

Citric Acid ($3HO,C_{12}H_5O_{11}$).

440. (C) When heated, citric acid at first melts, and is subsequently decomposed, emitting pungent fumes, which may be distinguished by their smell from those formed by tartaric acid under similar circumstances. A carbonaceous residue remains.

441. (C) *Chloride of calcium* ($CaCl$), when added to concentrated solutions of neutral citrates, gives a white precipitate of citrate of lime ($3CaO,C_{12}H_5O_{11}$), which is insoluble in potash, but soluble in chloride of ammonium. If this solution be boiled, the citrate of lime reprecipitates. Free citric acid gives no precipitate with this test; and the precipitate is not produced in moderately dilute solutions except on boiling.

442. (C) *Lime water* (CaO) fails to produce a precipitate in a cold solution, but if the mixture be boiled, citrate

of lime is thrown down, being less soluble in hot water than in cold.

443. Citric acid and the soluble citrates, when present in solutions containing peroxide of iron, alumina, and some other metallic oxides, prevent their precipitation by ammonia, owing to the formation of soluble double salts.

444. Citric acid, when heated with strong *sulphuric acid*, is decomposed; carbonic acid and carbonic oxide are given off with effervescence, and after some time, sulphurous acid (SO_2) is formed, and the mixture becomes dark colored.

Benzoic Acid ($HO,C_{14}H_5O_3$).

445. (C) When heated in a tube, this acid sublimes and condenses in the form of beautiful feathery crystals; the vapor has a peculiar aromatic odor, and causes an unpleasant sensation in the throat, inducing coughing.

446. *Perchloride of Iron* (Fe_2Cl_3) gives in neutral solutions a light yellowish-brown precipitate of perbenzoate of iron ($Fe_2O_3,3C_{14}H_5O_3$), which is soluble in acids, and is decomposed by ammonia.

447. (C) A mixture of *chloride of barium, ammonia, and alcohol* gives no precipitate with benzoic acid and the benzoates.

448. (C) When the solution of an alkaline benzoate, as benzoate of ammonia ($NH_4O,C_{14}H_5O_3$), is treated with strong *sulphuric* or *hydrochloric acid*, it is decomposed, and the liberated benzoic acid, being almost insoluble in water, is separated in the form of a white crystalline precipitate; while the sulphate of ammonia remains in solution.

$$NH_4O,C_{14}H_5O_3+HO,SO_3=NH_4O,SO_3+HO,C_{14}H_5O_3.$$

Gallic Acid ($3HO,C_{14}H_3O_7$).

449. When heated on platinum, gallic acid is charred, and emits a peculiar odor. The acid is sparingly soluble in cold, but dissolves readily in hot water.

450. (C) *Alkalies* mixed with solution of gallic acid, cause it to become brown immediately, by absorption of oxygen from the air.

451. (C) *Sesquichloride of iron* produces a bluish-black precipitate.

Tannic Acid ($C_{54}H_{22}O_{34}$).

452. This acid is also charred when heated on platinum. It dissolves readily in cold water.

453. The addition of an alkali causes a solution of tannic acid to become brown when exposed to the air.

454. (C) *Sesquichloride of iron* produces a bluish-black precipitate.

455. (C) *Dilute sulphuric acid* throws down a white precipitate, which is an insoluble compound of the two acids.

CHAPTER VII.

ORGANIC ALKALOIDS.

The organic alkaloids which are most likely to be met with in ordinary analysis are

Morphine, $C_{34}H_{19}NO_6$.
Strychnine, $C_{42}H_{22}N_2O_4$.
Quinine, $C_{40}H_{24}N_2O_4$.
Cinchonine, $C_{40}H_{24}N_2O_2$.

SECTION I.

Morphine.

A solution of the hydrochlorate ($C_{34}H_{19}NO_6,HCl+6Aq$) may be used.

456. *Concentrated nitric acid* produces, in solutions containing morphine, an orange color, appearing at once in pretty concentrated solutions, but only after a time, or on boiling, if the solution be dilute.

457. Neutral *perchloride of iron*, with neutral solutions of morphine, gives an inky blue or green color.

458. *Carbonate of soda* causes the separation of the

morphine in the form of a crystalline precipitate, especially if the solution be briskly stirred and allowed to stand; if this be collected on a filter, washed with a few drops of cold water, and moistened with neutral perchloride of iron, the inky color is very distinct.

(Opium. *Perchloride of iron* produces the fine red color due to the meconate of iron, which is not bleached by perchloride of mercury.

Acetate of lead causes a white precipitate of meconate of lead ($3PbO,C_{14}HO_{11}$)).

SECTION II.

Quinine.

A solution of the sulphate ($C_{40}H_{24}N_2O_4,HO,SO_3+7Aq$), in dilute sulphuric acid, may be used.

459. *Carbonate of soda* produces a bulky precipitate of hydrated quinine ($C_{40}H_{24}N_2O_4+6Aq$), which shrinks, after a time, and becomes crystalline; if the precipitate be suspended in water, and treated with chlorine, it passes through various shades of violet and red.

460. *Chlorine water*, followed by *ammonia*, produces a blue or green color in solutions of quinine.[1]

SECTION III.

Cinchonine.

461. In order to detect the presence of cinchonine in sulphate of quinine, ten grains of the salt are placed in a test-tube with two drachms of washed ether, and twenty drops of solution of ammonia are added. The mixture is agitated violently, and set aside for a minute or two, when it will be found to separate into two layers, the lower one being an aqueous solution of the sulphate of ammonia, and the upper layer, an ethereal solution of

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[1] **Quinidine may be distinguished from quinine by the solubility of its sulphate. The two sulphates may be readily separated by washing the mixture with a saturated solution of sulphate of quinine, which dissolves only the sulphate of quinidine.**

quinine; cinchonine being insoluble in both, will be seen at the surface of the lower layer.

SECTION IV.

Strychnine.

462. If a minute particle of strychnine be moistened, on a piece of white porcelain, with concentrated *sulphuric acid*, and touched with a drop of solution of *bichromate of potassa* upon the end of a glass rod, a deep blue color will be produced, afterwards changing to violet and red.

463. A similar change of color is observed when concentrated *nitric acid* is poured upon strychnine, and a few particles of the *peroxide* (brown oxide) *of lead* (PbO_2) are added.

464. If a solution of strychnine in an acid (hydrochloric) be mixed with *ammonia*, the precipitate which forms at first is redissolved by a slight excess of ammonia, and if the solution be allowed to stand, the strychnine crystallizes in distinct needles.

PART III.

QUALITATIVE ANALYSIS OF SUBSTANCES, THE COMPOSITION OF WHICH IS UNKNOWN.

CHAPTER I.

465. THE ultimate object of qualitative analysis is, of course, to ascertain the nature of the elements composing any substance presented for examination, without any clue whatever afforded by its origin or history. A system of analysis adapted to this purpose must be so framed that if all the non-metallic and metallic elements were present in the same substance, none should interfere with the detection of the others. Such a method is of necessity somewhat complicated, and would involve a great loss of time if applied to substances containing only one metal and one non-metallic element, or one base and one acid. Cases frequently present themselves in which it may be inferred, with little chance of error, that the substance to be examined is of this description, and the first division of this part of the work will therefore be devoted to the systematic course for the analysis of such substances, which will be found a very necessary introduction to the higher branch of analysis in which the most complex combinations are dealt with.[1]

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[1] In the appendix, there will be found a list of salts, &c., which may be taken for practice in qualitative analysis. The student should first examine a few of each kind with the assistance of the book, until he finds himself tolerably familiar with the processes; after which he may try them without reference to the printed directions.

QUALITATIVE ANALYSIS OF SUBSTANCES CONTAINING ONLY ONE METAL AND ONE NON-METAL, OR ONE BASE AND ONE ACID.

SECTION I.

Examination of a Solid Substance.

466. Observe and note down[1] the appearance of the substance, whether it is **crystalline** or **amorphous**; its **form** and **color**; especially whether it has any **metallic appearance.**[2] In the latter case pass on to (482).

467. PLACE A FRAGMENT OF THE SUBSTANCE IN A SMALL TUBE OF GERMAN GLASS, CLOSED AT ONE END: HEAT IT FIRST OVER A LAMP, AND AFTERWARDS IN THE FLAME OF THE BLOWPIPE.

(*a*) **It undergoes no change, and emits no odor or vapor.**

Absence of *water*, *organic matter*, &c. Pass on to (468).

(*b*) **It fuses, without any other apparent change.**

Probable absence of all metals except *potassium*, *sodium*, *barium*, *strontium*, *calcium*, and *aluminum*. Absence of *organic matter*. Pass on to (468).

(*c*) **It volatilizes entirely.**

Probable absence of all metals except *ammonium*, *mercury*, and *arsenic*. The probability is stronger if the volatilization takes place without the aid of the blowpipe. Also, absence of *boron* and *silicon*.

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[1] I cannot too strongly insist once more on the importance of making careful and accurate notes of all the experiments and observations which are made; they are not only often absolutely necessary for reference in the subsequent stages of the analysis, but the practice is also of the greatest value to the student, in cultivating habits of correct observation and facility of expression; besides at the same time impressing the facts more strongly on his recollection (6).

[2] In examining minerals and metals, it is advisable to take their specific gravity, when, by reference to a table (such as that in Dr. Thomson's Mineralogy, vol. i. p. 710), we are able to exclude those bodies which differ widely in their specific gravities from the substance under examination.

(*d*) It emits **brown sulphurous fumes,** condensing in **yellow globules** upon the side of the tube.
Presence of *sulphur.*

(*e*) It is **charred** and emits some **peculiar odor.**
Presence of *an organic substance.*

(*f*) **Cyanogen is evolved** (burning with a pink flame).
Presence of a *cyanide* or *ferrocyanide.*

(*g*) **Brown nitrous fumes** are evolved.
Presence of a *nitrate.*[1]

468. POUR A LITTLE STRONG SULPHURIC ACID UPON THE POWDERED SUBSTANCE IN A TEST-TUBE, and heat gradually, nearly to boiling, testing the odor and combustability of any gas evolved.

(*a*) **No evolution of gas or vapor** (except that of sulphuric acid); and **no carbonization;** probable absence of all acids except *sulphuric, phosphoric, boracic, silicic, arsenious, arsenic, antimonic,* and *stannic.* Pass on to (469).

(*b*) **Effervescence,** even without heat; probably *carbonic acid* (nearly inodorous), *sulphurous acid* (smell of burning sulphur), *hydrosulphuric acid* (smell of putrid eggs, indicating a sulphide), or *hypochlorous acid* (smell of chlorine). Pass on to (469).

(*c*) **Pungent suffocating acid fumes** are evolved; probably *hydrochloric acid* (indicating a chloride), *hydrofluoric acid* (indicating a fluoride, the tube found to be corroded, after washing and drying), *nitric acid* (often accompanied by brown vapors).

(*d*) **Violet vapors** are evolved; presence of *iodine.* Pass on to (470).

(*e*) **The substance becomes yellow or red,** and a **yellow chlorous gas** is evolved; presence of *chloric acid* (detonation often occurs on heating). Pass on to (469).

(*f*) **Combustible gas** (carbonic oxide) is evolved;

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[1] The student is reminded that the systematic examination by the blowpipe (141 *et seq.*) will be of great service in confirming the results obtained by qualitative analysis.

probably *oxalic acid*, *hydrocyanic acid* (indicating a cyanide), *hydroferrocyanic acid* (indicating a ferrocyanide; the mixture assumes a blue color), *citric acid*, or *tartaric acid* (carbonization).

(*g*) Vapors of **acetic acid** are evolved; the substance is an *acetate*. **Benzoic acid** vapors would be known by their odor.

(*h*) **Carbonization takes place**; presence of some *organic substance* other than the acids above named. (*Tartaric*, *gallic*, and *tannic* acids would be carbonized by sulphuric acid).

469. HEAT A LITTLE OF THE POWDER WITH DILUTE HYDROCHLORIC ACID, observing the odor of the gas, if any, evolved.

(*a*) The gas is **nearly inodorous**; probably *carbonic acid* (effervescence), confirm by (417). Pass on to (470).

(*b*) The gas has the **odor of burning sulphur**; presence of *sulphurous* or *hyposulphurous* acid; confirm by (366) and (369). Pass on to (470).

(*c*) The gas has the **odor of putrid eggs**; presence of a *sulphide;* confirm by (372). Pass on to (470).

(*d*) The gas has the **odor of chlorine**; presence of an *indifferent metallic oxide* (*e. g.*, the peroxides of manganese and lead), of *chloric acid*, of *hypochlorous acid*, or possibly of *nitric acid*. Pass on to (470).

(*e*) An **odor of hydrocyanic acid** is evolved; presence of a *cyanide* or a *ferrocyanide* (blue or green color).

To Dissolve the Substance for Analysis.

470. Place about ten grains of the powdered substance in a test-tube, and pour upon it two or three fluid drachms of distilled WATER; shake them well together, and if the substance is not dissolved, heat to boiling.

(*a*) The substance is **entirely dissolved.** Pass on to (485).

(*b*) **A great portion** of the substance **is dissolved.** Filter the solution, and examine it by (485).

(*c*) The substance is **not dissolved** to any considerable extent. Pass on to (471).

471. Boil a fresh portion of the substance (or, if it be scarce, the residue left in (470)) with DILUTE HYDROCHLORIC ACID.[1]

(*a*) It is **entirely dissolved.** Pass on to (513).

(*b*) **A great portion** of it **is dissolved.** Filter the solution, and examine it by (513).

(*c*) It is **not dissolved** in any considerable quantity. Pass on to (472).

472. Boil a fresh portion of the substance with DILUTE NITRIC ACID.

(*a*) It is **entirely dissolved.** Pass on to (512).

(*b*) **A great portion** of it **is dissolved.** Filter the solution and examine it by (512). Ascertain if the residue presents the characters of *sulphur*, indicating a sulphide (374).

(*c*) It is **not dissolved** to any great extent. Pass on to (473).

473. Boil a fresh portion with STRONG HYDROCHLORIC ACID, and add STRONG NITRIC ACID, very carefully, drop by drop.

(*a*) The substance is **dissolved.** Pass on to (513).

(*b*) **A great portion** of it **is dissolved.** Filter the solution and examine it by (513). Note whether the residue is sulphur (374).

(*c*) The substance is **undissolved.** Pass on to (474).

Analysis of a single Compound insoluble in water and acids.

474. Should the substance remain undissolved after the above course of treatment, heat a small portion of it upon a piece of platinum foil, or of porcelain, or in a porcelain crucible.

(*a*) It glows and **burns away,** emitting little or no smell; it is *carbon* (and of course is black).

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[1] Some substances, especially peroxide of iron, will dissolve only after long digestion with pretty strong hydrochloric acid.

(*b*) It **fuses** and burns with a **blue flame,** emitting sulphurous acid; it is *sulphur.*

(*c*) It **fuses to a brown drop,** without further change; it is probably *chloride of silver*, and will dissolve when heated with AMMONIA.

(*d*) **It undergoes none of these changes.** Pass on to (475).

475. Examine the substance by (400) to see if it is *fluor spar* (CaF).

476. Examine it before the blowpipe, on a platinum wire moistened with hydrochloric acid, to see if it is *sulphate of baryta* (green flame) or *sulphate of strontia* (crimson flame).[1]

477. Examine it by fusing on charcoal with carbonate of soda, or better, with cyanide of potassium, to see if it is *antimonic acid* (119), the *binoxide of tin* (118), or a compound of *lead* (115).

478. If it be found to contain lead, boil a small portion with ACETATE OF AMMONIA (made by mixing ammonia with a slight excess of acetic acid); if it be *sulphate*, or *chloride of lead*, it will be dissolved, and the solution may be tested for sulphuric acid with chloride of barium (364), for chlorine with nitrate of silver (384), and for lead with chromate of potash (329). If undissolved by acetate of ammonia, it is probably *silicate of lead.* Confirm by (138*a*).

479. Fuse it with a bead of borax, on platinum wire, to see if it is sesquioxide of chromium (131), or sesquioxide of iron (135).

480. Should the above experiments have failed to convince the analyst of the nature of the insoluble substance, it must be reduced to an impalpable powder (which is best effected in an agate mortar), mixed with three or four times its weight of dry CARBONATE OF SODA (or better, of carbonate of potash and soda (717), and thoroughly fused, on platinum foil, or in a platinum crucible,[2] with

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[1] Sulphate of lime might also occur here, although it may generally be dissolved by boiling with hydrochloric acid; the analyst must, therefore, not be too hasty in inferring the presence of strontia from the color of the flame (128).

[2] Great care is necessary in using a platinum crucible, that nothing

the aid of the blowpipe (641), until it remains in a perfectly tranquil state of fusion.

The fused mass is then allowed to cool, and heated with water for some time (the platinum foil being immersed in the water).

(*a*) The mass is **entirely dissolved** by the water; absence of all metals except *potassium*, *sodium*, *aluminum*, *chromium*, *manganese*, *zinc*, and *tin*. Mix the solution with an excess of HYDROCHLORIC ACID, evaporate it to dryness, heat the residue with dilute hydrochloric acid, filter the solution from the separated *silica* (414), and examine it for aluminum, chromium, manganese, zinc, and tin, as in (513 *et seq.*). If none of these be present, a fresh portion of the original substance must be examined for *potassium* and *sodium* by (540).

(*b*) **A portion** of the mass **remains undissolved**; the solution is filtered off (saving the residue for examination by (481), and acidulated with HYDROCHLORIC ACID; one portion being tested for *sulphuric acid* with CHLORIDE OF BARIUM, the other for *silicic acid* as in (414).

481. The undissolved residue is washed several times with water, dissolved off the filter in hot DILUTE HYDROCHLORIC ACID, and the solution examined for the metal according to (513).

is heated in it which is likely to corrode it. Compounds of the easily reduced and fusible metals, as tin, antimony, lead, bismuth, &c., substances containing sulphur, as metallic sulphides, caustic alkalies, nitrohydrochloric acid, besides many other substances, are all more or less injurious. When a platinum crucible is heated in a furnace or open fire, it must be placed in a covered earthen crucible to protect it from injury; a little pounded magnesia should be interposed between them, to prevent their sticking together, as at a high temperature the surface of the earthenware is liable to fuse.

SECTION II.

Examination of a simple substance having a metallic appearance.

482. A fragment of the substance is boiled for some time with DILUTE NITRIC ACID.

(*a*) It is **entirely dissolved.** Pass on to (512).
(*b*) It is **dissolved, with separation of sulphur** (374). Filter the solution, and examine by (512).
(*c*) A considerable **white residue** is left. This residue is probably either *antimonic acid*, *binoxide of tin*, or *sulphate of lead.* Pass on to (484).
(*d*) The substance remains **unaltered.** It is probably either *gold*, *platinum*, *carbon* in the state of graphite,[1] or *iodine* (in which case, **violet vapors** will be evolved). Examine by (483).

483. A small portion of this residue, or of the original substance, is very strongly heated on platinum foil.

(*a*) It **burns slowly away**; *graphite.*
(*b*) It remains **unaltered**; *gold* or *platinum.* Dissolve it in a mixture of hydrochloric and nitric acids, dilute the solution and add protochloride of tin; gold will give a purple-brown precipitate, and platinum a dark red color.

484. Boil another fragment of the substance with STRONG HYDROCHLORIC ACID.

(*a*) It rapidly **dissolves, evolving sulphuretted hydrogen** (372). Presence of *sulphide of antimony.* Filter the solution, and test for antimony (306). Confirm by (119).
(*b*) It **dissolves** slowly with effervescence, but **no** escape of **sulphuretted hydrogen.** Metallic *tin.* Test the solution with PERCHLORIDE OF MERCURY (345).
(*c*) It is **not dissolved** by hydrochloric acid, metallic *antimony* or *sulphide of lead.* Add a few drops

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[1] The graphitoid forms of boron and silicon would also remain unaltered.

of STRONG NITRIC ACID. Antimony will entirely dissolve, and may be tested for by (306). Sulphide of lead will be converted into the sparingly soluble chloride and sulphate, with separation of sulphur. Add an equal bulk of water, boil, filter, and test for lead with DILUTE SULPHURIC ACID (327).

SECTION III.

Examination of a liquid containing only one metal and one non-metal, or one base and one acid.[1]

PRELIMINARY EXPERIMENTS.

485. Carefully observe the **color, taste,** and **smell** of the liquid.

486. Place a drop upon a slip of thin glass, and evaporate it to dryness at a gentle heat.

(*a*) **No residue** is left upon the glass; if the liquid is colorless, tasteless, and neutral to test-paper, it is simply water.

(*b*) **A residue** is left; notice its appearance, and the action of heat upon it, making the inferences stated in (467 *c*, *d*, and *e*).

487. Try the action of the liquid upon blue and red litmus paper.

(*a*) The solution is **neutral** or **acid**; pass on to (490) or (512).

(*b*) The solution is **alkaline**; pass on to (488).

488. Mix a little of the alkaline solution, in a test-tube, with enough DILUTE NITRIC ACID to render it distinctly acid. If **no change** is produced, pass on to (490).

(*a*) **Effervescence** takes place, with evolution of a nearly inodorous gas; presence of an *alkaline carbonate;* pass on to (490).

(*b*) The liquid becomes **milky**; if the milkiness does not disappear on adding more nitric acid and gently heating, it is probably sulphur, and indicates the sulphide or hyposulphite of a metal of the first or second class. Test for a *sulphide* with acetate of lead (371), and for a *hyposulphite* with hydrochloric acid (369), in separate fresh portions of the original solution. Examine for the metal by (495).

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[1] Exclusive of any acid which the analyst may have employed to dissolve the substance under examination.

(*c*) A **distinct precipitate** separates, which is not dissolved by more nitric acid; this may be *stannic acid*, *arsenious acid*, *antimonic acid*, *silicic acid*, *benzoic acid*, or *uric acid*, which must have been in combination with an alkali in the original solution. Filter off the precipitate; test the solution for *ammonia* by (192), for *potash* by (186), and for *soda*, after having neutralized with a little potash or carbonate of potash, by (190). Pass on to (489).

489. Mix the original alkaline solution with a considerable quantity of HYDROSULPHURIC ACID, and acidulate it with HYDROCHLORIC ACID.

(*a*) A **yellow precipitate** is produced; add AMMONIA; if the precipitate dissolves, *arsenious acid* was present; if it does not dissolve, *stannic acid*. Confirm by (282, 118).

(*b*) An **orange precipitate** is produced; presence of *antimonic acid*. Confirm by (308).

(*c*) **No precipitate,** or only a **white precipitate** is produced. Acidulate another portion of the original alkaline solution with HYDROCHLORIC ACID, and add AMMONIA in excess; a **white gelatinous precipitate,** undissolved by the ammonia, is *silicic acid*. *Benzoic* and *uric* acids would be dissolved by the ammonia.[1]

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[1] Benzoic acid may be recognized by its feathery appearance, and by its dissolving readily on boiling the solution in which it was precipitated, emitting its peculiar odor. Uric acid would be identified by mixing the original solution with excess of nitric acid, evaporating just to dryness, and treating the (yellow or red) residue with ammonia, which would develop the pink color of the murexide.

CHAPTER II.

ACTUAL ANALYSIS OF SINGLE COMPOUNDS[1] IN A STATE OF SOLUTION.

SECTION I.

THE SUBSTANCE IS KNOWN TO BE DISSOLVED IN WATER.[2]

Examination for the Metal.

Hydrochloric Acid Test.

490. To a small portion of the solution, in a test-tube, add DILUTE HYDROCHLORIC ACID.

(*a*) **No precipitate** is produced. Pass on to (492).
(*b*) **A precipitate** is produced. Add an excess of hydrochloric acid, and if this dissolves it pass on to (492). But if the precipitate remains undissolved, it is probably either *chloride of silver*, *subchloride of mercury*, or *chloride of lead.*

491. In order to distinguish between them, a portion of the liquid containing the precipitate is shaken with excess of AMMONIA.

(*a*) The **precipitate is dissolved**; the metal present is *Silver.*
(*b*) The **precipitate becomes dark colored**; the metal present is *Mercury*, in the form of suboxide (Hg_2O), or some corresponding compound.
(*c*) The **precipitate remains undissolved**; the metal present is *Lead*, confirm by (325) and (327).

Hydrosulphuric Acid Test, in the same portion of the solution.

492. To the same portion to which hydrochloric acid

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[1] Including all simple salts, such as sulphate of potash, chloride of copper, &c.

[2] This may always be inferred when the liquid does not taste strongly acid.

has been added, add HYDROSULPHURIC ACID[1] in quantity at least equal to the volume of the solution employed, and until a distinct smell of the gas is perceived at the mouth of the tube after closing it with the thumb and shaking violently.

(*a*) **No precipitate** is produced. Pass on to (493).

(*b*) A **milkiness** only is produced, so that, on looking through the fluid, no distinct particles of precipitate are visible.[2] This is due to sulphur separated from the hydrosulphuric acid, and indicates the presence of some oxidizing agent, such as peroxide of iron, chromic acid, manganic or permanganic acid, nitrous acid, or chloric acid. Sulphurous acid would also give rise to separation of sulphur. Pass on to (493).

(*c*) A **black or brown precipitate** is produced. This may be due to the presence of *Lead*, *Copper*, *Bismuth*, *Mercury*, or *Tin*.[3]

To prove which of these it is, add to separate portions of the original solution, in test-tubes, the following tests, until one of them is found to indicate the metal present.

(1) DILUTE SULPHURIC ACID; if this causes a **white heavy granular precipitate,** *Lead* is present. Confirm by (329).

(2) AMMONIA; if this gives a rich **blue solution,** the metal is *Copper*. Confirm by (335).

(3) POTASH; if this gives a **yellow precipitate,** *Mercury*[4] is present in the

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[1] Smell the hydrosulphuric acid before using it; unless it smells very strongly it is useless (688), and must be again saturated with the gas. If it be preferred, the gas (687) may be passed through the solution itself, previously diluted with at least an equal volume of water.

[2] If there be any doubt, pass the liquid through a filter.

[3] Gold and platinum would also give a black or brown precipitate. The former would be precipitated as a purplish powder (from the original solution) by protosulphate of iron. Platinum would give a yellow precipitate with chloride of ammonium.

[4] If mercury were present in the form of cyanide, it would not be precipitated by potash. If this be suspected, the original solution

form of oxide (HgO), or of some corresponding compound. Confirm by (319, 322, and 424).

(4) Add to a little of the original solution, very carefully, AMMONIA, until a white precipitate is formed, which does not disappear on shaking; redissolve this by adding DILUTE HYDROCHLORIC ACID drop by drop, using as little as possible, and dilute the solution with eight or ten times its volume of water; a **milkiness** indicates *Bismuth.*[1]

(5) To a fresh portion of the original solution, add PERCHLORIDE OF MERCURY; the production of a **white or gray precipitate** indicates *Tin* in the state of protoxide (SnO) or some corresponding compound. Confirm by (344).

(*d*) The **precipitate caused by hydrosulphuric acid is yellow.** Either *Tin* or *Arsenic* is present.[2]

To determine which of these it is, add to a portion of the original solution, a slight excess of AMMONIA; if this causes a **white precipitate**, *Tin* is present in the form of binoxide[3] (SnO_2), or some corresponding compound (confirm by 347); while, if the

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may be acidulated with hydrochloric acid, and mixed with excess of protochloride of tin, which would give a gray precipitate of metallic mercury. The cyanogen must also be specially tested for, by decomposing the solution with an excess of yellow hydrosulphate of ammonia, filtering, evaporating nearly to dryness, and adding perchloride of iron (430).

[1] This test may be more accurately applied by evaporating the solution in hydrochloric acid almost to dryness in a test-tube or a small dish, and diluting largely with water.

[2] Cadmium would also furnish a yellow precipitate, distinguished from the sulphides of arsenic and tin by its insolubility in sulphide of ammonium.

[3] If the original solution be acid, the tin is probably present as bichloride, but if it be alkaline, the stannic acid is probably combined with an alkali; examine for the alkali as in the analysis of complex substances.

solution remains clear, *Arsenic* is probably present, as arsenious acid[1] or some corresponding compound. Confirm by (283).

(*e*) The **precipitate produced by hydrosulphuric acid is orange.** Presence of antimony, either as oxide (SbO_3), or antimonic acid[2] (SbO_5). Confirm by (305, 308).

493. If **hydrosulphuric acid has caused no precipitate, or one of sulphur only,** in the cold solution, heat the same portion, still smelling strongly of hydrosulphuric acid, to boiling for a minute or two.

(*a*) **No precipitate, or none but sulphur,** is produced. Pass on to (494).

(*b*) A **yellow precipitate** is produced. Either *Tin* (as binoxide or some corresponding compound) or *Arsenic*[3] (as arsenic acid (AsO_5), or some corresponding compound) is present. Proceed exactly as in (492*d*), confirming the presence of arsenic acid by the nitrate of silver test (298).

Hydrosulphate of Ammonia Test, in a fresh portion of the solution.

494. To a fresh portion of the original solution, add CHLORIDE OF AMMONIUM,[4] and AMMONIA in excess, so that it may be distinctly smelt at the mouth of the tube, after the latter has been closed by the thumb, and violently shaken. If a precipitate is produced, examine its appearance (white and gelatinous, alumina; dingy green, oxide of chromium or protoxide of iron; red-brown, per-

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[1] If the original solution be alkaline, the arsenious acid is combined with an alkali, which should be sought in the liquid from which the arsenic has been separated; as in the analysis of complex substances.

[2] If the original solution be alkaline, it probably contains antimoniate of potash, and will give a crystalline precipitate with carbonate of soda (190).

[3] If the original solution be neutral or alkaline, the arsenic acid is probably combined with an alkali, which must be tested for by (491).

[4] To prevent the precipitation of magnesia.

oxide of iron), but pass on, notwithstanding, and add HYDROSULPHATE OF AMMONIA.

(*a*) **No precipitate** is produced. Pass on to (495).

(*b*) A **black precipitate** is produced. Either *Iron*, *Nickel*, or *Cobalt* is present.

To distinguish between them, add to a fresh portion of the *original solution*, POTASH in excess.

(1) **Dingy green precipitate,** becoming rusty when exposed to air, indicates *Iron* as protoxide (FeO), or some corresponding compound. Confirm by (244).

(2) **Rust-colored precipitate,** *Iron* as peroxide (Fe_2O_3), or some corresponding compound. Confirm by (249).

(3) **Pale green precipitate** by potash; *Nickel.* Confirm by the blowpipe test, dipping the borax-bead into the solution (136).

(4) **Light blue precipitate,** becoming dirty pink or brown when boiled; *Cobalt.* Confirm by (133, 264).

(*c*) The **precipitate by hydrosulphate of ammonia is not black**; it indicates either *Aluminum*, *Chromium*, *Zinc*, or *Manganese.*

To distinguish between them, add to a fresh portion of the *original solution* AMMONIA in excess.

(1) **Pure white gelatinous semi-transparent precipitate,** insoluble in excess, and not tinged brown by exposure to air; *Aluminum.* Confirm by (217).[1]

(2) **Bluish-green precipitate**; *Chromium* in the form of oxide of chromium (Cr_2O_3). Confirm by (221).

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[1] If the original solution was alkaline, it is possible that silica may be mistaken for alumina in this part of the analysis. For the method of distinguishing silica, see (414).

(3) **White precipitate, becoming brown when exposed to air;** *Manganese* in the form of oxide (MnO). Confirm by the blowpipe (137), dipping the bead into the solution.

(4) **No precipitate, or a precipitate soluble in excess;** add to the ammoniacal solution a single drop of HYDROSULPHATE OF AMMONIA; if this causes a white precipitate, the metal is *zinc.* Confirm by (231). If the precipitate is greenish gray, it indicates *Chromium,* in the form of chromic acid[1] (CrO_3). Confirm by (227). If the precipitate is flesh-colored, it indicates *Manganese,* either in the form of oxide (MnO), sesquioxide (Mn_2O_3), manganic acid[2] (MnO_3), or permanganic acid (Mn_2O_7). (See footnote to (233).) Confirm by the blowpipe (137), dipping the bead into the solution.

Carbonate of Ammonia Test, in a fresh portion of the solution.

495. To a fresh portion of the original solution, add CHLORIDE OF AMMONIUM,[3] AMMONIA in excess, and CARBONATE OF AMMONIA. Heat gently.

(*a*) **No precipitate** is produced. Pass on to (497).

(*b*) A **white precipitate** is produced. Either *Barium, Strontium,* or *Calcium* is present.

496. To distinguish between them, add to a fresh portion of the original solution, SULPHATE OF LIME.

(*a*) A **slight precipitate is produced at once;** presence of *Barium.* Confirm by (130, 200).

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[1] The further examination for the base with which the chromic acid is united must be proceeded with, as in (550).

[2] The examination for the base with which this acid is united must be proceeded with as in the analysis of complex substances (550).

[3] To prevent the precipitation of magnesia.

(*b*) **No precipitate** is produced at once; allow the solution to stand aside for a few minutes, and if it still remains clear, apply heat. If a precipitate is formed, either on standing, or heating, *Strontium* is present. Confirm by (129). But if there is still no precipitate, *Calcium* is present. Test a fresh portion of the original solution with AMMONIA and OXALATE OF AMMONIA, which should give a white precipitate.

Phosphate of Soda Test, in the same portion of the solution.

497. To the solution in which carbonate of ammonia has failed to produce a precipitate, add PHOSPHATE OF SODA, and rub the sides of the tube, under the liquid, with a glass rod (212).

(*a*) **No precipitate** is produced. Pass on to (498).
(*b*) A **white crystalline precipitate** is produced. *Magnesium* is present.

Testing for the metals of the Alkalies, in fresh portions of the solution.

498. To a portion of the *original solution*, in a test-tube, add excess of POTASH and boil; if the **smell of Ammonia** is perceptible, *Ammonium* is present. Confirm by (193).

499. If ammonium be not detected,[1] place a little of the original solution in a watch glass, or upon a slip of glass, and add a drop of HYDROCHLORIC ACID and of BICHLORIDE OF PLATINUM; stir for a few seconds with a glass rod. A **yellow crystalline precipitate** indicates *Potassium.*

500. If there be no potassium, take a little more of the original solution upon a watch glass or slip of glass, dip a blue litmus paper into it, and if it be acid, add a drop of potash or carbonate of potash to render it alkaline; then add ANTIMONIATE OF POTASH and stir briskly with

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[1] If *arsenious acid*, *antimonic acid*, *stannic acid* (binoxide of tin), or *silicic acid*, has been discovered, the examination for potassium and sodium should be conducted as in the examination of complex compounds.

a glass rod. A **white crystalline precipitate** indicates *Sodium.*

Examination for the Non-metallic Element or Acid.[1]

501. To a portion of the original solution, add DILUTE SULPHURIC ACID, and heat gradually to boiling.

(*a*) **Effervescence** takes place, even in the cold, and a nearly *inodorous gas* is evolved; presence of *Carbonic Acid.* Confirm by (417).

(*b*) The **odor of burning sulphur** is perceptible; presence of *Sulphurous* or *Hyposulphurous Acid* (in the latter case, sulphur will separate).

(*c*) The gas has an **odor of putrid eggs;** presence of *Hydrosulphuric Acid* or of *Sulphur* combined with a metal.

(*d*) The **smell of chlorine** is perceived; presence of *hypochlorous* or of *chloric acid.* Confirm by (387, 391).

(*e*) **Hydrocyanic Acid is smelt** at the mouth of the tube; presence of *hydrocyanic acid* or a *cyanide*, or of *hydroferrocyanic acid* or a *ferrocyanide.* Confirm by (429, 433).

(*f*) *Acetic* and *benzoic acids* will be recognized by their characteristic odors.

Chloride of Barium[2] *Test.*

502. To a portion of the original solution, add enough AMMONIA to render it slightly alkaline, filter off any pre-

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[1] After determining the metal or base contained in a compound which we know to be soluble in water, it is of course unnecessary, in the subsequent examination, to look for any non-metal or acid which forms an insoluble compound with that metal or base. (Refer to the Table of Solubility in the Appendix.)

The following acids, which would have been detected in the examination for metals, may now be left out of consideration—viz., *Stannic, Chromic, Arsenious, Arsenic, Antimonic, Manganic,* and *Permanganic Acids.*

[2] If lead, silver, or mercury (as suboxide) be present in the solution, either nitrate of baryta must be substituted for the chloride of barium, or those metals must be precipitated by chloride of sodium, the precipitate filtered off, and the clear solution tested with chloride of barium.

cipitate which may be formed, and test the filtered solution with CHLORIDE OF BARIUM.

(*a*) **No precipitate** is formed. Pass on to (508).
(*b*) A **white precipitate** is produced. Probable presence of *sulphuric*, *phosphoric*, *boracic*, *oxalic*, *silicic*, *hydrofluoric*, *tartaric*, or *citric* acid.

503. To distinguish between these acids, add DILUTE HYDROCHLORIC ACID in excess, to the liquid containing the precipitate.

(*a*) The **precipitate disappears.** Pass on to (504).
(*b*) The **precipitate does not dissolve.** Presence of *sulphuric acid.*[1]

Chloride of Calcium Test.[2]

504. To a portion of the original solution, rendered alkaline by ammonia, and filtered if necessary, add CHLORIDE OF CALCIUM.

(*a*) **No precipitate** is produced. Pass on to (507).
(*b*) A **white precipitate** is produced. Probable presence of *Phosphoric*, *Boracic*, *Oxalic*, *Silicic*, *Hydrofluoric*, *Tartaric*, or *Citric* acid.

505. To distinguish between these acids, add CHLORIDE OF AMMONIUM to the liquid containing the precipitate.

(*a*) The **precipitate dissolves;** presence of *Boracic*, *Tartaric* or *Citric* acid. Test for boracic acid by (406), for tartaric acid by (437, 438), and for citric acid by (441, 442) in the original solution.
(*b*) The **precipitate does not dissolve;** presence of *Phosphoric*, *Silicic*, *Oxalic*, or *Hydrofluoric* acid.

506. To distinguish between these, treat the precipitate with ACETIC ACID.

(*a*) The **precipitate dissolves;** presence of *Phosphoric* or *Silicic* acid; add to this solution a drop of

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[1] A little sulphuric acid may be expected as an impurity in most commercial salts.

[2] If lead, silver, or mercury (as suboxide) be present, pass on to (508).

PERCHLORIDE OF IRON; a **yellowish-white precipitate** indicates *Phosphoric acid.* Confirm by (381).

Test for silicic acid by (414) in the original solution.

(*b*) The **precipitate does not dissolve** in acetic acid; presence of *Oxalic* or *Hydrofluoric* acid. If the precipitate be **opaque and pulverulent,** it indicates *Oxalic Acid.* Confirm by (422). If it be nearly **transparent and gelatinous,** it indicates *Hydrofluoric Acid* or a *Fluoride.* Confirm by (399).

507. Should no precipitate be produced by chloride of calcium in the cold, the solution must be boiled; if this causes **a precipitate,** *Citric Acid* is probably present. Confirm by (441, 442).

If there be **no precipitate,** pass on to (508).

Perchloride of Iron Test.[1]

508. To a portion of the original solution, add PERCHLORIDE OF IRON.

(*a*) **No change** is observed. Pass on to (509).

(*b*) **Blue precipitate**; presence of *Hydroferrocyanic Acid* or a *Ferrocyanide.*

(*c*) **Light buff precipitate**; presence of *Benzoic Acid.* Confirm by (448).

(*d*) **Black precipitate**; presence of *Gallic* or *Tannic* acid. Test the solution by (450, 455).

(*e*) **Red color** without precipitate; presence of *Acetic Acid.* Confirm by (427).

Nitrate of Silver Test.

509. To a fresh portion of the original solution, add enough dilute nitric acid to render it slightly acid, and NITRATE OF SILVER.

(*a*) **No precipitate** is produced. Pass on to (511).

(*b*) **A precipitate** is produced; presence of either *Hydrochloric*, *Hydriodic*, or *Hydrocyanic* acid.

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[1] If lead, silver, or mercury (as suboxide) be present, they must be removed by adding chloride of ammonium and filtering before applying this test.

510. To distinguish between these, allow the precipitate to subside, wash it by decantation, and shake it with AMMONIA in excess.

(*a*) The **precipitate dissolves**; either *Hydrochloric* or *Hydrocyanic* acid is present. To a little of the ammoniacal solution, add excess of STRONG NITRIC ACID and boil; if the **precipitate at first produced redissolves,** *Hydrocyanic Acid* or a *Cyanide*[1] is present. Confirm by (429).

If the nitric acid produces a **permanent precipitate,** *Hydrochloric Acid*[2] or a *Chloride* is present.[3] Confirm by (386).

(*a*) The **precipitate does not dissolve** in ammonia; even on agitation and gently warming. Presence of *Hydriodic Acid* or an *Iodide.* Confirm by (393).

Special Tests for Nitric and Chloric Acids.

511. If the acid has not yet been discovered, either there is none present, or it is nitric or chloric acid.

(*a*) BOIL A LITTLE OF THE ORIGINAL SOLUTION WITH STRONG SULPHURIC ACID AND COPPER FILINGS; if **brown fumes** are given off, *Nitric Acid* is present.[4] Confirm by (361).

(*b*) If the acid is not nitric, test another part of the original solution for *Chloric Acid* by boiling with STRONG SULPHURIC ACID (387).

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[1] Cyanide of mercury would not respond to the nitrate of silver test (note to 492).

[2] A little chloride may be expected as an impurity in most commercial salts.

[3] Hydrobromic acid would behave in a similar way, but would be recognized in testing according to (386), by the very irritating brown fumes of bromine.

[4] Nitrous acid (NO_3) might here be mistaken for nitric acid; but the nitrites would disengage red fumes when heated with dilute sulphuric acid.

SECTION II.

THE SUBSTANCE IS DISSOLVED IN AN ACID.[1]

Examination for the Metal.

Hydrochloric Acid Test.[2]

512. Add to the solution a little DILUTE HYDROCHLORIC ACID.

(*a*) **No precipitate** is produced. Pass on to (513).

(*b*) **A precipitate** is produced. Probable presence of *Silver*, *Mercury* (as suboxide Hg_2O), or *Lead*. Examine the precipitate by the directions given in (491).

Hydrosulphuric Acid Test, in the same portion of the solution.

513. Mix the solution with a large excess of a saturated solution of HYDROSULPHURIC ACID.

(*a*) **No precipitate** is produced. Pass on to (514).

(*b*) A **milkiness** only is produced, so that, on looking through the fluid, no distinct particles of precipitate are visible.[3] This is due to sulphur separated from the hydrosulphuric acid. Pass on to (514).

(*c*) A **black or brown precipitate** is produced. Presence of *Lead*, *Copper*, *Bismuth*, *Mercury*[4] (as oxide HgO), or *Tin*. (See note to 402*c*.)

Test for these metals in separate portions of the original solution, as directed in (492*c*.)

(*d*) A **yellow precipitate** is produced. Either Tin[5]

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[1] If the analyst be uncertain upon this head, the taste of the solution will determine it.

[2] This may of course be omitted when hydrochloric acid has been employed in preparing the solution.

[3] If there be any doubt, pass the liquid through a filter.

[4] When nitric acid has been employed in preparing the solution, any mercury which had originally existed as suboxide or some corresponding compound, would have been converted into the oxide.

[5] If nitric acid has been employed in dissolving the substance, it may have converted the protoxide of tin into binoxide. A similar remark applies to antimony and arsenic.

(as binoxide or some corresponding compound) or *Arsenic* is present. Distinguish between them by the tests given in (492*d*). (See note to 492*d*.)

(*e*) An **orange precipitate** is produced. Presence of *Antimony*, either as oxide (SbO_3) or as antimonic acid (SbO_5).[1] Confirm by (305, 308).

514. If **hydrosulphuric acid has caused no precipitate** or one of sulphur only, in the cold solution, heat the same portion to boiling, and should this fail to give a precipitate, saturate the hot liquid with hydrosulphuric acid gas (687), and again boil.

(*a*) **No precipitate,** or none but sulphur, is produced. Pass on to (515).

(*b*) A **yellow precipitate** is produced. Either *Tin* (as binoxide or some corresponding compound), or *Arsenic* (as arsenic acid (AsO_5), or some corresponding compound) is present. Distinguish between them by the tests given in (492*d*).

Hydrosulphate of Ammonia Test, in a fresh portion of the solution.

515. To a fresh portion of the original solution, add CHLORIDE OF AMMONIA,[2] and AMMONIA in excess, so that it may be distinctly smelt at the mouth of the tube, after the latter has been closed by the thumb, and violently shaken. If a precipitate is produced, examine its appearance (white and gelatinous, alumina, silica, or an insoluble phosphate; pulverulent, an insoluble oxalate; crystalline, phosphate of magnesia and ammonia; dingy green, oxide of chromium or protoxide of iron; red-brown, peroxide of iron), but pass on, notwithstanding, and add HYDROSULPHATE OF AMMONIA.

(*a*) **No precipitate** is produced. Pass on to (516).

(*b*) A **black precipitate** is produced. Either *Iron*,[3] *Nickel*, or *Cobalt* is present.

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[1] If *Stannic Acid* (SnO_2), *Antimonic Acid* (SbO_5), *Arsenious* or *Arsenic Acid* be detected, the further examination for the base must be conducted as in the analysis of complex substances.

[2] To prevent the precipitation of magnesia as hydrate.

[3] If nitric acid has been used in dissolving the substance, the solution will contain the iron as peroxide (Fe_2O_3), or some corresponding

Distinguish between them by the tests given in (494*b*).

(*c*) The **precipitate** by hydrosulphate of ammonia **is not black**; either *Aluminum*, *Chromium*, *Zinc*, *Manganese*, *Phosphate* or *Oxalate* of *Baryta*, *Strontia*, or *Lime*, *Phosphate of Magnesia* or *Silica* is probably present.

To distinguish between them, add gradually to a fresh portion of the original solution POTASH in excess.

(1) **Green precipitate,** soluble in excess to a green liquid, and precipitated on boiling; presence of Chromium as oxide (Cr_2O_3).

(2) **White precipitate, insoluble in excess, becoming brown** on exposure to air; presence of *Manganese*, Confirm by (137).

(3) **White precipitate, soluble in excess**; presence of *Aluminum*, *Zinc*, or *Silica*.

To distinguish between them, acidify the potash solution with HYDROCHLORIC ACID, and add AMMONIA in excess.

If there be **no precipitate,** or a very trifling one (from impurity of the potash) *Zinc* is present, as may be confirmed by adding SULPHIDE OF AMMONIUM to the ammoniacal liquid.

If a **flocculent precipitate** was produced by the excess of ammonia, either *Aluminum* or *Silica*, or both, must be present.[1] Evaporate a portion of the original acid solution to dryness, in a porcelain dish, and heat the residue with DILUTE HYDROCHLORIC ACID,

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compound, in whatever form it existed in the original substance. Iron previously existing as ferrocyanogen might also occur here, from the breaking up of that radical by the action of acids.

[1] Silica being an acid.

which will leave any *Silica*[1] undissolved, to be collected on a filter and tested by the blowpipe (138*a*).

Mix the hydrochloric solution with enough POTASH to redissolve the precipitate produced at first, and acidify the liquid strongly with ACETIC ACID. A white gelatinous precipitate will be *phosphate of Alumina.* If there be no precipitate, add excess of AMMONIA, which will throw down the *Alumina.*

(4) Potash has produced, in the original solution, a **white precipitate, insoluble in excess, and not becoming brown** by exposure; presence of a *Phosphate* or *Oxalate of Baryta, Strontia,* or *Lime,* or *Phosphate of Magnesia.*

To distinguish between them, add to a portion of the original solution, AMMONIA until a permanent precipitate is formed, and ACETIC ACID in excess. If the precipitate be dissolved, it is a *Phosphate* (confirm by 378); if undissolved, an *Oxalate* (confirm by 422).

To ascertain the presence of *Baryta* or *Strontia,* test the original solution with SULPHATE OF LIME, as directed in (496).

If these be absent, and the compound has been found to be an oxalate, it must be *Oxalate of Lime;* but if it be a phosphate, it may be either *Phosphate of Lime* or *Phosphate of magnesia.*

To ascertain which, add to a portion of the original solution AMMONIA till a permanent precipitate is formed, then ACETIC ACID till the precipitate is redissolved, and OXALATE OF AMMONIA; a white precipitate indicates *Lime.* If there be no precipitate, add excess of AMMONIA, and rub the sides of the tube with a glass rod; a crystalline precipitate indicates *Magnesia.*

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[1] If silica be detected, the further examination for the base must be conducted as in the analysis of complex substances (590).

(5) Potash has produced **no precipitate** in the original acid solution. Probable presence of chromium as *Chromic Acid.*[1] Confirm by (223).

Carbonate of Ammonia Test, in the same portion of the solution.

516. To the same portion of the solution in which hydrosulphate of ammonia has failed to give a precipitate, add CARBONATE OF AMMONIA, and heat gently.

(*a*) **No precipitate** is produced. Pass on to (517).

(*b*) **A precipitate** is produced. Presence of *Barium, Strontium,* or *Calcium.*

Distinguish between them by the tests mentioned in (496), applied to fresh portions of the original solution.

Phosphate of Soda Test, in a fresh portion of the solution.

517. To a fresh portion of the original solution, add CHLORIDE OF AMMONIUM, AMMONIA in excess, and PHOSPHATE OF SODA, and rub the sides of the tube, under the liquid, with a glass rod (212).

(*a*) **No precipitate** is produced. Pass on to (518).

(*b*) A **white crystalline precipitate** is produced. *Magnesium* is present.

Testing for the Metals of the Alkalies, in fresh portions of the solution.

518. The salts of the alkali-metals being, as a general rule, soluble in water, a substance requiring an acid to dissolve it is not likely to contain potassium, sodium, or

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[1] **If chromic acid be discovered, the base in combination with it must still be detected by further examination.**

ammonium, unless antimonic acid or silicic acid be present; in which case the examination for those metals should be conducted as in the analysis of complex substances.

If the analyst has not had the opportunity of testing the solubility of the original substance in water, the examination for potassium, sodium, and ammonium should be conducted as in (498).

Examination for the Non-Metallic Element or Acid.[1]

A. *The analyst has a portion of the solid substance at his disposal.*

519. The experiments described in (467, 468, and 469) should be very carefully made, and will establish the presence or absence of *Sulphurous Acid*, *Hypochlorous Acid*, *Hydrochloric Acid* (a chloride), *Hydriodic Acid* (an iodide), *Acetic Acid*, *Benzoic Acid*, *Carbonic Acid*, *Hydrofluoric Acid* (a fluoride), and *Nitric Acid.*

The same experiments would also lead to a correct conclusion as to the absence of *Tartaric Acid*, *Citric Acid*, *Gallic Acid*, *Tannic Acid*, *Oxalic Acid*, *Hydrocyanic Acid*, (a cyanide), *Hydroferrocyanic Acid* (a ferrocyanide).

During the experiments to dissolve the substance (470), it will have been ascertained whether a *Sulphide* is present or not.

520. Dissolve a portion of the substance by boiling with DILUTE HYDROCHLORIC ACID, filter the solution, if necessary, and examine by the following tests:—

(*a*) CHLORIDE OF BARIUM; a **white precipitate** indicates *Sulphuric Acid.*[2]

(*b*) AMMONIA in excess, and HYDROSULPHATE OF AMMONIA; heat the solution to boiling, and filter, if necessary; to the filtered liquid, add a clear mixture of SULPHATE OF MAGNESIA, CHLORIDE OF AMMONIUM, and AMMONIA; rub the sides of the tube, under the liquid, with a glass rod. **Crystalline precipitate**; presence of *Phosphoric Acid.*[1]

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[1] Assistance may now be derived from the Table of Solubilities (Appendix). See note to (501). Besides the acids therein enumerated, *silicic acid* need not now be sought, as it would have been already detected (515*c* 3); *chloric acid* and *hyposulphurous acid* cannot exist in the acid solution.

[2] It must be remembered that a slight precipitate is probably due to an impurity in the substance.

(*c*) Dip a piece of TURMERIC PAPER into the solution, and dry it at a gentle heat; **brown orange color** indicates *Boracic Acid.* Confirm by (407).

521. Boil a portion of the original substance with a strong solution of CARBONATE OF SODA for a few minutes; the base will be thus converted into an insoluble carbonate, and the acid will pass into solution. Filter, neutralize the solution carefully with NITRIC ACID.

(*a*) Add PERCHLORIDE OF IRON to detect *Hydroferrocyanic*, *Tannic*, and *Gallic* Acids. See (508).

(*b*) Acidify the solution very slightly with dilute nitric acid; heat it to expel free carbonic acid, render it very slightly alkaline with ammonia, and add CHLORIDE OF CALCIUM to detect *Oxalic*, *Tartaric*, and *Citric Acids.* See (504).

(*c*) Test for *Hydrocyanic Acid* (a cyanide) by (429).

522. Boil a portion of the original substance with DILUTE NITRIC ACID, filter, if necessary, and add NITRATE OF SILVER, to test for *Hydrochloric Acid* (a chloride). See (509).

B. *The analyst has only the acid solution at his disposal.*[2]

523. Dilute a portion of the solution with water, add DILUTE HYDROCHLORIC ACID, filter, if necessary, and add CHLORIDE OF BARIUM; **white precipitate,** not dissolved by much water, *Sulphuric Acid.*

524. To another portion of the solution[3] add NITRATE OF SILVER.

(*a*) **No precipitate** is produced. Pass on to (525).

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[1] If this acid were in combination with baryta, strontia, lime, magnesia, or alumina, this test would not detect it, but it would then have been already detected in the examination for the metal (515).

[2] The presence of hydrosulphuric acid would be known by its odor.

[3] If hydrosulphuric acid be present, it should be expelled by a gentle heat before applying this test.

(*b*) **A precipitate** is produced. Presence of *Hydrochloric Acid*,[1] *Hydriodic Acid*, or *Hydrocyanic Acid.*

Examine as directed at (510).

525. Test for *Phosphoric* and *Boracic* acids, according to (520, *b* and *c*) in the original solution.

526. Test for *Nitric Acid*,[2] by (361).

527. To another portion of the original solution add a strong solution of CARBONATE OF SODA in excess, boil for a minute or two, filter, if necessary, neutralize the solution carefully with NITRIC ACID.

(*a*) Add PERCHLORIDE OF IRON to detect *Benzoic*, *Tannic*, *Gallic*, and *Acetic* Acids. See (508).

(*b*) Acidify the solution very slightly with DILUTE NITRIC ACID, heat it to expel free carbonic acid, render it very slightly alkaline with AMMONIA, and add CHLORIDE OF CALCIUM to detect *Boracic*, *Oxalic*, *Hydrofluoric*, *Tartaric*, and *Citric* Acids. See (504).

527*a*. Test specially for *Hydrocyanic Acid* by (429), in another portion of the original solution.

CHAPTER III.

QUALITATIVE ANALYSIS OF SUBSTANCES WHICH MAY CONTAIN TWO OR MORE METALS AND NON-METALS, OR TWO OR MORE BASES AND ACIDS.

Introductory Remarks.

Unless we have reason to know that a substance intended for analysis contains only one base and one acid, it is necessary to assume that it *may* contain any or all of the more common saline compounds. Such an analysis is of course considerably more complicated than that of a single salt; and consequently the necessity of having a well-devised scheme of experiments is here even greater than in the former case, when only one base and acid had to be determined.

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[1] Which may have been used to dissolve the original substance.

[2] Which may have been used to dissolve the original substance.

The method of dealing with such a mixture is, first to separate the whole of the metals of the fourth class (if any are present) by passing hydrosulphuric acid gas through a solution of the substance acidified with hydrochloric acid, and filtering the solution from the precipitate: the precipitate is then dissolved in acid, and tested successively for each of the metals of the fourth class. The filtered solution, containing all the bases but those of the fourth class, is then neutralized, and treated with hydrosulphate of ammonia, which throws down all the metals of the third class (if any are present): and the precipitate filtered from the solution is dissolved in acid, and tested successively for each metal of the third class. The solution filtered from the sulphides can now only contain any metals of the first and second class that may be present, which may readily be distinguished by a few simple tests.

The student must be careful when making these experiments, that he adds sufficient of the various reagents, to throw down *the whole* of the metals affected by them, since any traces of the metals belonging to a class supposed to have been entirely removed from the solution, would materially interfere with the indications afforded by the subsequent tests. For example, in the analysis of a mixture of a salt of lead and a salt of lime, if sufficient hydrosulphuric acid were not passed through the solution to separate the whole of the lead, a black precipitate of sulphide of lead would be formed on the addition of hydrosulphate of ammonia to the filtered liquid, indicating the presence of one or more metals of the third class, none of which are really present. On the other hand, the addition of a large excess of any of the reagents is also to be avoided, as being not only useless and wasteful, but in many cases mischievous.

Both these errors may be avoided by adding the reagents in small successive portions: and when the experimenter has reason to think that he has added sufficient, let him filter a few drops of the mixture, and apply to the solution a little more of the reagent: if this produces no further precipitate, he may conclude that enough has been added.

In the course of an analysis, especially of a complicated substance, it is often necessary to have several solutions in hand at the same time; to avoid confusion, each of these should be labelled with a bit of gummed paper, with a letter or mark upon it, referring to a corresponding letter in the note book (6).

As one portion of the substance to be analyzed has to be carried through several operations, it is advisable that the quantity operated on should not be very small. When the substance is a solid, twenty or thirty grains may be used; and when in solution, an ounce or two (according to the degree of concentration) will be found a convenient quantity.

SECTION I.

EXAMINATION OF A SOLID SUBSTANCE.

528. The substance should first be examined according to the directions given in (466-469), but the analyst must be more careful in drawing his conclusions from the indications presented, on account of the complication of phenomena arising from the presence of more than one compound.

The process of dissolving the substance also requires modification.

To dissolve the substance for analysis.

(If the substance has a *metallic appearance*, pass on to (541).

529. Boil about 20 grains of the powdered substance, in a test-tube or small flask, with two or three fluid drachms of distilled water for a minute or two.

(*a*) **The substance is entirely dissolved.** Pass on to (546A).

(*b*) **An insoluble residue is left.** Set the tube aside for a few minutes, to allow the residue to settle. Pour the liquid upon a filter (leaving the residue in the tube, if possible), and reserve the solution for examination according to (546A). Wash the residue once or twice with water, by decantation (634), if possible, or upon the filter (not allowing the washing waters to mix with the solution first filtered), and proceed as in (530).[1]

530. Boil the residue in a tube or flask, with about a drachm of STRONG HYDROCHLORIC ACID (unless *silver* or *lead* be suspected,[2] when nitric acid must be employed) for a minute or two,[3] add about an equal quantity of water, and again boil.

(*a*) **The substance is entirely dissolved.** Pass on to (588).

(*b*) **An insoluble residue is left.** Add a few drops of STRONG NITRIC ACID (or of hydrochloric if nitric acid was employed before), and again boil. Should this fail to dissolve it, add more water, boil, and filter, reserving the clear solution for examination by (588). Wash the residue upon the filter as long as the washings are acid to litmus paper, dry it (631), and treat it according to (531).

Analysis of a complex compound, insoluble in water and acids.[4]

531 (*a*) **The insoluble substance is black.** Heat a small portion of it upon a piece of platinum foil; if it burns entirely away it contains nothing but *carbon*, and perhaps *sulphur*, which may be recognized by its odor;

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[1] If the residue has been collected upon a filter, it may either be washed through the perforated filter with as little water as possible, or it may be dried as at (631) and carefully detached.

[2] From the preliminary blowpipe examination, see foot-note to (467*g*).

[3] If red oxide of iron be present, prolonged digestion with hydrochloric acid is often necessary.

[4] The chief substances which would remain undissolved after the above treatment are the *sulphates of baryta*, *strontia* (and possibly of *lime* and *oxide of lead*), *chloride of silver*, *silicic acid*, and the *silicates* of any of the *metallic oxides*, *alumina* and *oxide of chromium* in certain states, *fluoride of calcium*, *binoxide of tin*, *antimonic acid*, *chrome-iron ore*, *sulphur*, *charcoal*.

but if any incombustible residue be left, the substance must be examined according to (532).

(*b*) **The insoluble substance is not black.** Heat a small portion upon a piece of platinum foil, to ascertain whether it contains any *sulphur;* if any incombustible residue be left, examine the original insoluble substance according to (532).

532. Fuse a small portion of the insoluble substance on charcoal, with carbonate of soda, before the blowpipe (115 *et seq.*) and examine very carefully, to see if it contains any *lead*, *tin*, *silver*, or other easily reducible metal.

(*a*) **No metal** is obtained. Pass on to (539).

(*b*) **A metallic globule** is obtained. Examine it carefully to see if any certain inference can be drawn from its malleability, &c., and proceed to examine the rest of the insoluble substance according to (533).

533. Boil the insoluble substance with a strong solution of ACETATE OF AMMONIA (prepared by mixing ammonia with an *excess* of acetic acid) for a few minutes.

(*a*) It is **entirely dissolved,** and consisted therefore of *sulphate of Lead.* Test the solution for sulphuric acid with chloride of barium, and for lead with chromate of potash (329).

(*b*) **A residue is left:** filter the solution and test it as in (*a*), for sulphate of lead. Wash the residue, and treat it as in (534).

534. Heat a small portion of the residue on platinum foil.

(*a*) **It burns entirely away,** and consists therefore either of *sulphur* (known by the odor), or of *carbon* (known by the color), or of both.

(*b*) An **incombustible residue** is left. Pass on to (535).

535. The insoluble residue from (533*b*) is heated with strong AMMONIA.

(*a*) It is **entirely dissolved,** and was therefore *chlo-*

ride of silver. Reprecipitate by nitric acid, and confirm by the blowpipe (117).

(*b*) **A residue is left:** filter the solution, and test it, as in (*a*) for chloride of silver. Wash the residue, and treat it by (536).

536. Heat a small portion of the residue on platinum foil.

(*a*) It **burns entirely away,** and consists therefore of *sulphur* or *carbon*, or of both.

(*b*) An **incombustible residue** is left. Dry the whole of the insoluble residue from (535*b*), and heat it to dull redness in a porcelain crucible (640) until all the sulphur and carbon, if any be present, are burnt off; then carefully examine a part of the residue on charcoal with carbonate of soda, to see if it still contains any reducible metal. If no metal be found, pass on to (539), but otherwise, proceed as follows.

537. The residue is reduced to a very fine powder, and about three-fourths of it intimately mixed with about four times its weight of CARBONATE OF SODA (or better, of the carbonate of potash and soda, see (717), and fused in a porcelain[1] crucible over a blowpipe lamp, being retained in a fused state for several minutes. After cooling, the crucible and its contents are placed in water, and digested with it, at a moderate heat, with occasional stirring, until the fused mass is entirely detached from the crucible, when the latter may be removed. When the fused mass has been thoroughly disintegrated, the solution is filtered and examined for metals, according to (546), and for non-metals and acids according to (573*b*).[2] The residue on the filter is well washed until the washings are no longer alkaline, some hot DILUTE NITRIC ACID is poured over it, and should this fail to dissolve it, it should be rinsed into a tube and boiled with the acid.

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[1] Since alumina and silica would be derived from the porcelain, these must be specially tested for according to (538).

[2] In ordinary cases, only *sulphuric*, *silicic*, *hydrochloric*, and *hydrofluoric* acids need be sought for in this solution.

(*a*) The residue is **entirely dissolved.** Examine the solution for metals according to (588), except for potassium and sodium, which must be sought for by the method described at (540).

(*b*) An **insoluble residue** is left. This should be washed once or twice, and boiled with STRONG HYDROCHLORIC ACID (the hydrochloric solution being examined for metals by (588)), and should this fail to dissolve it, the residue, which consists of some substance (such as chrome-iron ore), not attacked by the alkaline carbonates, should be dried and fused with a mixture of equal weights of carbonate of soda and nitrate of potash, in a porcelain crucible, being kept in fusion for half an hour. On treating the mass with water, the solution, which is yellow, if chromium be present, may be examined for acids by (573*b*), and for metals (except potassium and sodium) by (546). The residue, after washing, may be digested with strong hydrochloric acid, which will now at least partly dissolve it,[1] and the solution may be examined for metals by (589). The remaining fourth of the incombustible residue from (536) must now be examined for alumina and silica in the following manner.

538. About 50 grains of pure HYDRATE OF POTASH are fused in a silver crucible over a spirit or Argand gas-lamp (not over a gauze-burner), and the substance in fine powder is added by degrees, the mass being retained in fusion until it is perfectly tranquil. The crucible is then allowed to cool and placed in water; when the fused mass is dissolved, the crucible is removed, the solution filtered, acidulated with nitric acid, and evaporated to dryness. The dry residue is heated with dilute nitric acid, which will leave *silica* undissolved to be tested by the blowpipe (138, 138*a*). The filtered solution is treated with sulphuretted hydrogen, to separate any of the metals of that class, filtered, evaporated to a small bulk, precipi-

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[1] Any portion still remaining insoluble must be tested before the blowpipe (142).

tated by an excess of ammonia, and the precipitate examined for alumina as in (566).

539. If the insoluble substance be not found to contain an easily reducible metal (532), it is reduced to a very fine powder,[1] and about three-fourths of it are fused with CARBONATE OF POTASH AND SODA as directed in (537), but the operation may be conducted in a platinum crucible, or capsule, or upon a piece of platinum foil turned up at the edges. The subsequent part of the process is conducted as in (537), but DILUTE HYDROCHLORIC ACID may be substituted for nitric in dissolving the residue from the filter. The acid solution is examined for metals, except potassium and sodium, by (589). The remaining fourth of the insoluble substance is examined for potassium and sodium in the following manner.[2]

Examination of insoluble silicates for potassium and sodium.

540. The finely powdered substance is intimately mixed with five or six times its weight of pure HYDRATE OF BARYTA,[3] and calcined, in a platinum crucible, for about half an hour. The mass is digested in water, acidulated with hydrochloric acid till it is thoroughly disintegrated, when the solution is poured into a dish, and evaporated to dryness. The dry residue is heated with water, and baryta-water is added till the solution has a strongly alkaline reaction. The precipitate (containing the silica, and all the metals present except those of the alkalies and alkaline earths) is filtered off, and the solution is mixed with ammonia and carbonate of ammonia, and heated, to precipitate the alkaline earths. The filtered solution is evaporated to dryness (a few drops of hydrochloric acid being added as the evaporation draws to a close, in order to insure the conversion of the potassium and sodium

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[1] Should any carbon or sulphur have been discovered in it (531), the substance should be calcined in an open crucible until these are burnt off.

[2] This is only necessary when silicic acid has been found to be a constituent of the substance.

[3] If lead be present in the substance, a little nitrate of baryta may be added to the mixture to prevent the reduction of lead and consequent damage to the crucible.

into chloride); the dry residue is then further treated as in (552).

Examination of a complex substance having a metallic appearance.

541. The substance is reduced to as fine a state of division as possible, and boiled with DILUTE NITRIC ACID, in a flask, as long as any action appears to take place; about an equal quantity of WATER is then added, and the mixture again boiled.

(*a*) The substance is **entirely dissolved.** Pass on to (588).

(*b*) **A residue is left;** The solution is filtered or poured off from the residue, and examined according to (542).

542. The residue is well washed, dried, and calcined in a porcelain crucible at a moderate heat, any indication of the presence of *sulphur* being carefully looked for.

(*a*) **The substance burnt entirely away.** It consisted of *sulphur* only.

(*b*) An **incombustible residue** is left. If this residue be **light-colored,** it may contain *binoxide of tin*, *antimonic acid*, or *sulphate of lead*, and should be examined by (544). If the residue be **dark-colored,** it may contain in addition to those substances, *gold*, *platinum*, and carbon in the state of graphite.

543. Boil this dark-colored residue with a mixture of HYDROCHLORIC ACID with one-third of NITRIC ACID.

(*a*) It is **entirely dissolved,** and therefore probably consisted of finely-divided *gold* or *platinum*, or both. Test one part of the solution for gold with PROTOCHLORIDE OF TIN (483*b*), and another for platinum with chloride of ammonium.[1]

(*b*) An **insoluble residue** is left. This may consist of *sulphate of lead*, *antimonic acid*, *binoxide of tin*

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[1] Some sulphate of lead would also be dissolved by the mixed acids, but need not be regarded.

(all **white**), and *carbon* as graphite.[1] If the residue is **black**, heat it to redness for some minutes upon a strip of platinum foil; if it burns entirely away, it is simply carbon; but should it be white, or leave an incombustible residue, treat it as follows:—

544. Boil with a strong solution of ACETATE OF AMMONIA (ammonia mixed with acetic acid in slight excess) for a few minutes.

(*a*) The substance is **entirely dissolved**; test the solution for *sulphate of lead* as in (533*a*).

(*b*) **A residue is left.** Filter the solution, and test it for sulphate of lead (533*a*). Dry the washed residue and mix it in a porcelain crucible with three or four parts of powdered CYANIDE OF POTASSIUM. Cover the crucible, heat it strongly till the contents are in a state of perfect fusion, and keep it so for ten minutes. When the crucible is cool, nearly fill it with water, and heat to boiling in order to dissolve the cyanate of potash and cyanide of potassium; allow the undissolved portion (possibly tin, antimony, and carbon) to subside, wash it by decantation,[2] rinse it into a test-tube and boil with a little STRONG HYDROCHLORIC ACID for a few seconds. Decant a little of the hydrochloric solution into another tube, and test for *Tin* with PERCHLORIDE OF MERCURY (345). To the rest of the acid (on the residue) add a drop or two of NITRIC ACID, and continue to boil. Any *Carbon* will remain undissolved. The acid solution is divided into two parts, the smaller of which is mixed with saturated HYDROSULPHURIC ACID when an orange precipitate will indicate the presence of *Antimony*, and a yellow or drab precipitate that of *Tin*. Should the color of the precipitate be doubtful, the other portion of the acid solution should be mixed with an excess of CARBONATE OF AMMONIA,[1] boiled, and filtered. The filtered solution is mixed with a drop of hydrosulphate of ammonia, to precipitate any traces of copper, &c., which may be filtered off, and then with a slight excess of DILUTE HYDROCHLORIC ACID, when the **orange precipitate** will be obtained if *Antimony* be present.

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[1] Some of the rarer metals, such as the alloy of osmium and iridium, might also be expected here, as might also titanic acid.

[2] If there be any metallic globules, try them as to their malleability. If they are brittle, it is well to pound them in a mortar before boiling with hydrochloric acid.

SECTION II.

EXAMINATION OF A LIQUID SUPPOSED TO CONTAIN TWO OR MORE METALS AND NON-METALS, OR BASES AND ACIDS.

545. Examine the liquid as directed in (485, 486, and 487).

(*a*) The solution is **neutral or acid;** pass on to (546A) or (588).

(*b*) The solution is **alkaline;** pass on to (546).

546. Mix a part of the alkaline solution with enough DILUTE NITRIC ACID to render it distinctly acid. If **no change** is produced, pass on to (546A).

(*a*) **Effervescence** takes place, with evolution of a nearly inodorous gas; presence of an alkaline carbonate; pass on to (546A).

(*b*) The liquid becomes **milky:** if the milkiness does not disappear on adding more nitric acid, and gently heating, it is probably sulphur, and indicates the presence of a *sulphide* or *hyposulphite* of a metal of the first or second class; boil, filter, and pass on to (588).

(*c*) A distinct **precipitate** separates, which is not dissolved by more nitric acid. Filter off the precipitate and examine the solution by (588). Wash the precipitate, and endeavor to dissolve it by boiling with strong hydrochloric acid and a few drops of nitric acid; filter the solution, and examine it by (588). If there be any undissolved residue, it must be washed, dried, and examined according to (431).

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[1] If the presence of tin has not yet been proved, it should be sought in the precipitate produced by carbonate of ammonia, by incinerating the filter containing it, fusing the ashes with cyanide of potassium, washing any reduced metal with water, boiling with hydrochloric acid, and testing with chloride of mercury for tin.

CHAPTER IV.

ACTUAL ANALYSIS OF COMPLEX COMPOUNDS IN A STATE OF SOLUTION.[1]

SECTION I.

THE SUBSTANCE IS KNOWN TO BE DISSOLVED IN WATER.[2]

Examination for Metals.

546A. Reserve about half of the solution to be examined for non-metallic bodies and acids.

To the other half, add DILUTE HYDROCHLORIC ACID.

(*a*) **No precipitate** is produced. Pass on to (547).

(*b*) **A precipitate** is produced. Add an excess of hydrochloric acid, and if this dissolves it, pass on to (547). But if the precipitate remains undissolved, even on heating, filter the solution, and reserve the precipitate for examination by (554).

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[1] When a complex compound has been partly dissolved by water, and partly by acids, it becomes a question whether the solution should be mixed and examined together. In general, it may be stated that when a knowledge of the arrangement of the constituents is desired, the solutions should be examined separately, but when the sole object is to learn what metals and non-metallic bodies are present, the solutions may be mixed, observing these precautions before mixing:—(1) Acidulate the aqueous solution with nitric acid, and examine any precipitate as in (546*c*); afterwards mix the clear liquid with hydrochloric acid, and reserve any precipitate to be examined by (554). (2) Mix the nitric solution with hydrochloric acid, and examine any precipitate by (554). (3) If, on mixing the acid solutions after these precautions, any precipitate be obtained, it must be collected and examined as an insoluble substance (532).

[2] This may always be inferred when the liquid does not taste strongly acid.

Hydrosulphuric Acid Test.

547. A few drops of the clear solution from (546) are mixed with a saturated solution of HYDROSULPHURIC ACID, and boiled.

(*a*) **No precipitate,** except sulphur (see 492*b*) is produced. Treat the remainder of the solution (to which no hydrosulphuric acid has been added) according to (549).

(*b*) **A precipitate** (which is not sulphur) is produced. The remainder of the clear solution from (546) is thoroughly saturated with hydrosulphuric acid gas, which must be passed through it (689) until, after removing the delivering tube, and blowing the air from the surface of the solution, the latter smells distinctly of the gas; a considerable quantity of a saturated solution of hydrosulphuric acid is then added, the solution heated to boiling and filtered, the precipitate being reserved for examination by (556).

548. The liquid filtered from (547) is mixed with more HYDROSULPHURIC ACID (which should give no precipitate if the former operation had been well executed) and boiled. If a **yellow precipitate** (due to to the presence of arsenic acid) appears, the boiling liquid must be treated with hydrosulphuric acid gas till the whole of the arsenic is precipitated. The yellow precipitate may be filtered off, and examined for arsenic by the tests given in (275, 277). The filtered liquid is examined by (549).

Hydrosulphate of Ammonia Test.

549. A small portion of the liquid in which hydrosulphuric acid gave no precipitate, or which has been filtered off from the precipitate, is tested with a few drops of hydrosulphuric acid, to be quite sure that nothing precipitable by that reagent is left, and is then mixed with CHLORIDE OF AMMONIUM,[1] AMMONIA in *excess*, and HYDROSULPHATE OF AMMONIA (disregarding any precipitate by ammonia alone), and gently heated.

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[1] To prevent the precipitation of magnesia.

(*a*) **No precipitate** is produced; proceed with the examination of the main portion of the solution by (550).

(*b*) **A precipitate** is produced; treat the whole of the solution in the same way, collect the precipitate upon a filter, and reserve it for examination by (561). Proceed to examine the filtered solution according to (550).

Carbonate of Ammonia Test.

550. A small portion of the solution in which hydrosulphate of ammonia gave no precipitate, or which has been filtered[1] off from the precipitate, is mixed with CHLORIDE OF AMMONIUM, AMMONIA in excess, and CARBONATE OF AMMONIA, and heated to boiling.

(*a*) **No precipitate** is produced; proceed with the examination of the solution by (551).

(*b*) **A precipitate** is produced; treat the whole of the solution in the same way, collect the precipitate upon a filter, and reserve it for examination by (567). Proceed to examine the filtered solution by (551).

Phosphate of Soda Test.

551. A small portion of the ammoniacal liquid in which carbonate of ammonia gave no precipitate, or which has been filtered off[2] from the precipitate, is mixed with PHOSPHATE OF SODA, well stirred with a glass rod (rubbed

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[1] If this filtered liquid is brown, it probably contains a little sulphide of nickel, and must be evaporated until the excess of sulphide of ammonium is expelled (known by the odor), slightly acidulated with dilute hydrochloric acid, the precipitate collected upon a filter and tested for nickel, and the filtered liquid mixed with ammonia and carbonate of ammonia. In some cases, when much organic matter is present, the filtrate from the hydrosulphate of ammonia precipitate may still contain iron, chromium, &c. In such a case it must be evaporated to dryness, calcined till the organic matter is consumed, the residue dissolved by digesting in hydrochloric acid, and the examination recommenced from (549).

[2] If this solution be very dilute, it should be concentrated by evaporation. It is also advisable to test it with a little oxalate of ammonia, to be quite sure that the baryta, strontia, and lime have been entirely removed.

against the sides of the tube), and set aside for some minutes.

(*a*) **No precipitate** is produced; evaporate the main portion of the solution (to which no phosphate of soda has been added) to dryness in a small porcelain dish, and calcine the residue as long as any fumes of ammoniacal salts are evolved; examine the residue by (552).

(*b*) A **crystalline precipitate** is produced; presence of *magnesium*; evaporate the main portion of the solution to dryness, calcine the residue as long as any fumes of ammoniacal salts are evolved, dissolve it in a little water, add BARYTA-WATER as long as it causes any precipitate (sulphate of baryta and hydrate of magnesia), heat, and filter; mix the filtered liquid with AMMONIA and CARBONATE OF AMMONIA, heat to boiling; filter off the precipitated carbonate of baryta, evaporate the filtered liquor to dryness, calcine the residue as long as any fumes are evolved, and examine it according to (552).

Tests for Potassium and Sodium.

552. The residue, after evaporation and calcination, is dissolved in as little water as possible, the solution filtered if necessary and divided into two parts, to be examined according to (*a*) and (*b*) respectively.

(*a*) One part of the clear solution is mixed, in a watch-glass, with a drop or two of DILUTE HYDROCHLORIC ACID to render it acid, and then with a few drops of BICHLORIDE OF PLATINUM, the mixture being well stirred with a glass rod and set aside for some time. A **yellow granular precipitate** indicates *potassium.*

(*b*) The other part of the solution is mixed, in a watch glass, with a few drops of ANTIMONIATE OF POTASH, and well stirred with a glass rod. A **white crystalline precipitate** indicates *sodium.*

Test for Ammonium.

553. A fresh portion of the original solution is boiled with an excess of POTASH, to see if **alkaline vapors** with the **odor of ammonia** are evolved, indicating the presence of *Ammonium.*

Examination of the Precipitate by Hydrochloric Acid.

554. Wash the precipitate once or twice with cold water (by decantation if possible), and boil it with a considerable quantity of WATER.

(*a*) It is **entirely dissolved,** and consisted only of chloride of lead, indicating the presence of *Lead.* Confirm by (327).

(*b*) It is **not entirely dissolved**; pour off, or filter off the clear solution, and test for lead with DILUTE SULPHURIC ACID (327). Examine the residue by (555).

555. Warm the residue gently with AMMONIA.

(*a*) It is **entirely dissolved,** and consisted only of chloride of silver, indicating the presence of *Silver;* confirm by reprecipitating the ammoniacal solution with excess of nitric acid.

(*b*) **A residue** is left; if the residue is **black** it is suboxide of mercury, and indicates the presence of *Mercury* as suboxide (Hg_2O) or some corresponding compound. If the residue be **white,** it is probably some chloride of lead. In either case, filter it off, and test the solution for *Silver* by adding an excess of NITRIC ACID, which would precipitate the chloride of silver.

Examination of the Precipitate by Hydrosulphuric Acid.

556. The precipitate is well washed, upon the filter, with water, until the washings are no longer strongly acid; a small portion of it is then taken on the end of a glass rod, and placed in a little yellow HYDROSULPHATE OF AMMONIA at the bottom of a test-tube; the mixture is heated to boiling.

(*a*) The precipitate is **entirely dissolved**; examine the main portion of the precipitate according to (557).

(*b*) **A residue** is left; filter the solution from the residue, and add to the clear liquid some DILUTE HYDROCHLORIC ACID in slight excess, and HYDROSULPHURIC ACID. If the precipitate thus produced be **white,** or at most slightly brown or gray, it consists only of sulphur separated from the hydrosulphate of ammonia by the hydrochloric acid (373), showing that the hydrosulphate had not dissolved any sulphide of tin, antimony, or arsenic, and the main portion of the original precipitate may then be examined according to (558).

But if the precipitate produced by hydrochloric and hydrosulphuric acids, in the hydrosulphate of ammonia solution, be **yellow** or **orange,** it contains the sulphide of *tin*, *antimony*, or *arsenic;* the main portion of the original precipitate must then be boiled with the yellow HYDROSULPHATE OF AMMONIA (being first rinsed into a test-tube or small flask[1]), and the liquid filtered from the undissolved portion, which is to be reserved for examination by (558). The filtered liquid is mixed with a slight excess of DILUTE HYDROCHLORIC ACID, some HYDROSULPHURIC ACID ADDED, the precipitate collected upon a filter, washed with water, and examined according to (557).

557. The precipitate is gently heated for some time with solution of SESQUICARBONATE OF AMMONIA (saturated in the cold).

(*a*) It is **entirely dissolved,** and consisted only of sulphide of arsenic; reprecipitate the solution with a slight excess of DILUTE HYDROCHLORIC ACID, and confirm the presence of *Arsenic*[2] by (277).

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[1] **If the quantity of the precipitate be very small, some hot hydrosulphate of ammonia may be poured over it upon the filter.**

[2] **Ascertain whether the arsenic existed as arsenious or arsenic acid by special tests applied to the original solution.**

(*b*) **A residue** insoluble in carbonate of ammonia is left. Filter it off, and test the solution for sulphide of arsenic as in (*a*); wash the residue upon the filter, and dissolve it in boiling HYDROCHLORIC ACID with the aid of two or three drops of NITRIC ACID (using as little acid as possible); mix the solution with CARBONATE OF AMMONIA in considerable excess, and boil it. If no precipitate is obtained, the absence of *Tin* may be inferred; but if a precipitate is produced, collect it upon a filter, wash it, and treat it as in the note to (544). In either case, test for *Antimony* in the solution by adding a drop of hydrosulphate of ammonia, filtering off any traces of sulphide of copper, &c., thus precipitated, and mixing the filtered liquid with excess of hydrochloric and hydrosulphuric acids, to obtain the orange precipitate.[1]

558. That portion of the precipitate which has been proved to be insoluble in hydrosulphate of ammonia, is washed (until the washings are no longer precipitated by nitrate of silver) and boiled with DILUTE NITRIC ACID.

(*a*) It is **entirely dissolved,** except a few light flakes of sulphur; examine the solution according to (559).

(*b*) **A residue** (beside sulphur) is left. Filter off the solution, and examine it by (559). Boil the residue with a little NITRO-HYDROCHLORIC ACID (three measures of hydrochloric and one of nitric acid), add a slight excess of AMMONIA, then a slight excess of HYDROCHLORIC ACID, and boil with two or three slips of BRIGHT COPPER to test for *Mercury* (324).

559. Evaporate the solution to as small a bulk as is convenient, mix it with DILUTE SULPHURIC ACID, and set it aside for some minutes. **A heavy granular precipitate** indicates the presence of *Lead;* filter the solution, and examine it according to (560).

560. To the solution in which sulphuric acid has failed

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[1] Special tests must be applied to the original solution to ascertain the states of oxidation in which the tin and antimony existed there.

to produce a precipitate, or which has been filtered from the precipitate, add AMMONIA in excess.

(*a*) **No precipitate** is produced, but the solution has a **blue color**, presence of *Copper*.

(*b*) A **white precipitate** is produced; filter it off; observe if the solution has the **blue color** indicating *Copper*, dissolve the precipitate in as small a quantity as possible of DILUTE HYDROCHLORIC ACID, evaporate it to the last drop, and dilute largely with water, a **milkiness** indicates *Bismuth.*

Examination of the Precipitate by Hydrosulphate of Ammonia.

561. The precipitate is well washed, upon the filter, till the washings are colorless, and dissolved in as small a quantity as possible of a mixture of three measures of DILUTE HYDROCHLORIC, and one measure of DILUTE NITRIC ACID, which may be heated and poured over the precipitate upon the filter. The solution is boiled, filtered, if necessary, from separated sulphur, and a small portion mixed with excess of AMMONIA.

(*a*) A **brown** or rusty **precipitate** is produced. Examine the main portion of the solution by (562).

(*b*) **No brown precipitate** is produced. Examine the main portion of the solution by (564).

562. The solution is mixed with AMMONIA, which is added by degrees, with constant agitation, until the precipitate at first produced is redissolved with some difficulty on agitation. A considerable quantity of ACETATE OF AMMONIA (prepared by mixing ammonia with acetic acid in slight *excess*) is then added, and the solution (which should now be distinctly acid) is diluted with water, and boiled for three or four minutes. The precipitate (containing *peroxide of iron*, and possibly *alumina* and *oxide of chromium*) is filtered off, and examined according to (566), the solution (a small portion of which should now give no brown precipitate with ammonia, if the iron has been entirely precipitated) being treated as directed in (563).

563. The solution is concentrated by evaporation, mixed with a slight excess of AMMONIA, and with a strong solution of HYDROSULPHURIC ACID (or the gas is passed through it).

(*a*) **No precipitate** is produced. Absence of *manganese*, *nickel*, *cobalt*, *aluminum*,[1] and *chromium*.

(*b*) **A precipitate** is produced. Collect it upon a filter, and examine it as follows:—

Treat the precipitate upon the filter with hot DILUTE HYDROCHLORIC ACID. (Should this leave any black residue, examine it with a borax-bead for nickel and cobalt before the blowpipe (133, 136); dissolve the remainder in nitric acid, and add it to the hydrochloric solution).

564. Boil the solution, allow it to cool, and mix it with POTASH in excess.

(*a*) **A precipitate** is produced. Collect it upon a filter, and examine it by (565). Examine the filtered solution as in (*b*).

(*b*) **No precipitate** is produced. Boil the solution for some time, filter off any precipitate of *Oxide of Chromium*, and test it before the blowpipe (131, 222). Acidify the filtered solution with HYDROCHLORIC ACID, and add AMMONIA *in excess;* any precipitate of *Alumina*[2] is to be filtered off, and the solution tested with HYDROSULPHATE OF AMMONIA, when a **white precipitate** will indicate *Zinc.*

565. The precipitate produced by potash (564*a*) is washed, and a little of it is tested with a bead of CARBONATE OF SODA, before the blowpipe, for *Manganese* (137). Dissolve it off the filter in as little warm DILUTE HYDROCHLORIC ACID as possible, and add CARBONATE OF

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[1] Aluminum and chromium have also to be looked for in the precipitate from (562).

[2] Since the potash generally contains a little alumina, it is well to test a bulk equal to that employed in the experiment, by mixing it with excess of hydrochloric acid, and afterwards with excess of ammonia.

AMMONIA in excess. Boil the solution, filter off any precipitate (of carbonate of manganese and oxide of chromium, to be tested as directed in (222)), and add CYANIDE of POTASSIUM to the solution as long as it produces any change in the liquid. Mix the solution with excess of HYDROCHLORIC ACID, and boil, with repeated addition of hydrochloric acid, till no more hydrocyanic acid is evolved; then add POTASH in excess, and boil, with repeated addition of potash, till no more ammonia is evolved.

(*a*) Collect the precipitate upon a filter, wash it, and test with a bead of borax, before the blowpipe, for *Nickel.*

(*b*) The alkaline solution, which may contain *cobalticyanide of potassium*, is acidulated with NITRIC ACID, evaporated to dryness, the residue fused for some minutes, allowed to cool, and heated with water to dissolve the potash-salts; the black residue is tested with a bead of borax, before the blowpipe, for *Cobalt.*

566. The precipitate produced by boiling with acetate of ammonia (562) is washed, dissolved off the filter in warm DILUTE HYDROCHLORIC ACID, and the solution boiled with excess of POTASH.

(*a*) **A precipitate** is produced. Filter it off, and examine the solution according to (*b*). Wash the precipitate, dissolve a small portion of it in HYDROCHLORIC ACID, and test with ferrocyanide of potassium for *Iron.*[1] Dry the remainder, and test for *Chromium*[2] by (222).

(*b*) **No precipitate** is produced. Acidify the solution with HYDROCHLORIC ACID, and add excess of AMMONIA to precipitate *Alumina.* See note to (564*b*).

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[1] Test specially in the original solution to ascertain whether the iron exists as protoxide or peroxide.

[2] The state of oxidation in which the chromium existed in the original solution must be ascertained by special tests.

Examination of the Precipitate by Carbonate of Ammonia.

567. The precipitate is washed, and dissolved off the filter in as little DI~~LUTE HYDROCHLORIC ACID~~ as possible. $C_4 H_4 C_4$

568. To a *small portion* of the solution add SULPHATE OF LIME.

(*a*) **A precipitate** is formed **at once.** Presence of *Barium.* Pass on to (569).

(*b*) **A precipitate** is formed only **on standing** for some time, or on gently heating. Presence of *Strontium.* Pass on to (569*c*).

(*c*) **No precipitate** is formed even on gently heating. Test *another portion* of the hydrochloric solution with AMMONIA and OXALATE OF AMMONIA for *Calcium.*

569. To the remainder of the hydrochloric solution, add an excess of ~~HYDROFLUOSILICIC ACI~~D, evaporate to dryness, and digest the residue with ALCOHOL, which will leave the silicofluoride of barium ~~undissolved~~. Filter the solution, ~~and again evaporate to dryness~~. Test a part of the residue on platinum wire, to see what color it imparts to the blowpipe flame (128, 129), dissolve the remainder in water and divide the solution into three parts. K [illegible]

(*a*) Test the smallest part with ~~HYDROFLUOSILICIC ACID~~ to ascertain that all the barium has been separated. K [illegible]

(*b*) Test the second part with SULPHATE OF LIME for *Strontium.*

(*c*) Mix the third portion with excess of SULPHATE OF SODA, allow it to stand for some time, filter off the precipitate, again test the solution with sulphate of soda, to be sure that all the strontium has been precipitated, and add OXALATE OF AMMONIA; a **white precipitate** indicates *Calcium.*

Examination for Non-Metals and Acids in complex substances known to be dissolved in Water.[1]

570. To a portion of the original solution, add DILUTE SULPHURIC ACID, and heat gradually to boiling.

(*a*) **Effervescence** takes place even in the cold, and a nearly inodorous gas is evolved; presence of *Carbonic Acid.* Confirm by (417).

(*b*) The **odor of burning sulphur** is perceptible; presence of *Sulphurous* or *Hyposulphurous Acid* (in the latter case, sulphur will separate).

(*c*) The gas has an **odor of putrid eggs**; presence of *Hydrosulphuric Acid* or of *Sulphur* combined with a metal.

(*d*) The **smell of chlorine** is perceived; presence of *Hypochlorous* or of *Chloric Acid.* Confirm by (387, 391).

(*e*) **Hydrocyanic Acid is smelt** at the mouth of the tube; presence of *Hydrocyanic Acid*, or a *Cyanide*, or of *Hydroferrocyanic Acid* or a *Ferrocyanide.* Confirm by (429, 433).

(*f*) *Acetic and Benzoic* acids will be recognized by their characteristic odors.

571. Test a part of the solution for *Nitric Acid* by (359, 361).

572. Test a part of the solution for *Sulphuric Acid* by mixing it with excess of HYDROCHLORIC ACID, filtering if necessary, and adding CHLORIDE OF BARIUM. **White precipitate** indicates *Sulphuric Acid.*

573. Test if the solution is acid or alkaline.

(*a*) The solution is **acid.** Add CARBONATE OF SODA till it is pretty strongly alkaline, boil and filter. Examine the filtered solution by (574).

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[1] It is of course impossible that such a solution can exist, containing *all* the bases and acids in the list, since several of them would form salts which are insoluble in water, as baryta and sulphuric acid, oxide of silver and hydrochloric acid, &c. It is consequently unnecessary, after having determined the bases in a mixture of salts soluble in water, to look for any acids which form with them salts that are insoluble. (See Table of Solubilities in the Appendix.)

(*b*) The solution is **alkaline**. Acidulate it *slightly* with DILUTE NITRIC ACID, heat gently for some time to expel any carbonic, sulphurous, or hydrosulphuric acid, filter, if necessary, from any precipitated sulphur, silicic, antimonic, or stannic acid,[1] add CARBONATE OF SODA till it is very slightly alkaline, boil, filter, if necessary, and proceed with the filtered solution as in (574).

574. To a *small part* of the solution which has been rendered alkaline by carbonate of soda, add a little HYDROSULPHATE OF AMMONIA, and boil.

(*a*) **No precipitate** is produced; proceed with the remainder of the solution according to (575).

(*b*) **A precipitate** is produced; saturate the remainder of the solution with HYDROSULPHURIC ACID gas, boil till there is no more smell of the gas, and filter. Examine the filtered liquid according to (575).

575. Mix the alkaline solution with DILUTE NITRIC ACID, in a dish, till it is *slightly* acid, heat gently, to expel the carbonic acid, and render the solution slightly alkaline with AMMONIA. Proceed to (576).

Chloride of Calcium Test.

576. To a portion of the slightly ammoniacal solution add CHLORIDE OF CALCIUM.

(*a*) **No precipitate** is produced. Pass on to (579).

(*b*) **A precipitate** is produced. This precipitate may possibly contain *Phosphoric*, *Oxalic*, *Tartaric*, *Boracic*, and *Arsenic* acids, in combination with lime, and *Fluoride* of calcium. Collect the precipitate upon a filter, and treat the solution as in (579). Examine the precipitate by (577).

577. Treat the precipitate with a little CHLORIDE OF AMMONIUM.

(*a*) It is **entirely dissolved**; only *Tartaric* and *Boracic* acids can be present. Test specially for both, in fresh portions of ammoniacal solution by (406, 407, 587).

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[1] Examine this precipitate before the blowpipe.

(*b*) It is **not entirely dissolved**; *Phosphoric*, *Arsenic*, and *Oxalic* acids may be present, as well as *Tartaric* and *Boracic* acids. Test specially for the two last, in fresh portions of the ammoniacal solution, according to (406, 407, 587). Examine the undissolved portion of the precipitate according to (578).

578. Treat the undissolved portion of the precipitate with ACETIC ACID.

(*a*) It is **entirely dissolved**; only *Phosphoric* and *Arsenic* acids can be present. Pass on to (580).

(*b*) It is **not entirely dissolved**; *Oxalic* and *Hydrofluoric* acids may be present. Test specially for both according to (399, 587).[1]

579. The solution in which chloride of calcium has failed to give a precipitate, or which has been filtered from the precipitate, is tested with a little more chloride of calcium, and boiled. If any precipitate is produced, *Citric Acid* is probably present. Confirm by (587*c*).

Perchloride of Iron Test.

580. Another portion of the ammoniacal solution (575) is gently heated in a dish till it is *no longer alkaline*, and PERCHLORIDE OF IRON is gradually added.

(*a*) **No precipitate** is produced. Pass on to (581).

(*b*) **A precipitate** is produced. It may indicate *Phosphoric* (yellowish white), *Arsenic* (yellowish white), *Hydroferrocyanic* (blue), *Benzoic* (buff), *Tannic* (black), and *Gallic* (black) acids. Filter off the precipitate, and pass on to (581).

581. If the solution in which perchloride of iron has produced no precipitate, or which has been filtered from the precipitate, has a **red color**, it may indicate *Acetic* or *Hydrosulphocyanic* acid. Add PERCHLORIDE OF MERCURY.

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[1] In general, the opaque and pulverulent character of the precipitate of oxalate of lime (insoluble in acetic acid) will sufficiently indicate oxalic acid.

(*a*) The **color disappears;** *Hydrosulphocyanic Acid* or a *Sulphocyanide* is present.

(*b*) The **color remains;** *Acetic Acid* is present. Confirm by (427).

Nitrate of Silver Test.

582. Another portion of the ammoniacal solution (575) is gently heated in a dish till it is *no longer alkaline*, and NITRATE OF SILVER is added, as long as any further precipitate is produced.

(*a*) **No precipitate** is produced. Pass on to *Special Testing for Acids* (585*a*).

(*b*) **A precipitate** is produced. This may indicate *Hydrochloric* (white), *Hydriodic* (yellow), *Hydrocyanic* (white), *Hydroferrocyanic* (white), *Phosphoric* (yellow), *Boracic* (white), *Arsenic* (brown), *Oxalic* (white), *Tartaric* (white), *Citric* (white), and *Benzoic* (white), acids.

583. Add to the liquid containing the precipitate, DILUTE NITRIC ACID.

(*a*) The **precipitate is dissolved** entirely. Pass on to *Special Testing for Acids* (585*a*.)

(*b*) An **insoluble residue** is left. This may be *Chloride*, *Iodide*, *Cyanide*, or *Ferrocyanide* of silver.

584. Wash the precipitate by decantation, and shake it with AMMONIA.

(*a*) It is **entirely dissolved;** either chloride or cyanide of silver; reprecipitate by DILUTE NITRIC ACID, allow the precipitate to subside; wash it by decantation, and boil with STRONG NITRIC ACID. *Chloride* of silver will remain **undissolved,** indicating the presence of *Hydrochloric Acid* or a *Chloride*. *Cyanide* of silver would be **dissolved** by the hot nitric acid. Test specially for Hydrocyanic Acid by (586).

(*b*) It is **not entirely dissolved;** either *Iodide* or *Ferrocyanide* of silver must be present. Ferrocyanogen will have been already detected (580). Confirm the presence of *Hydriodic Acid* by (393). Filter the solution from the precipitate, and examine the filtered liquid as in (*a*).

Special Testing for Acids which may have escaped detection in the preceding course.

585. *Phosphoric Acid.* Refer to the notes made during the examination for metals to see if any *Arsenic* was discovered.

(*a*) **No arsenic is present.** To a portion of the ammoniacal solution (575), add a mixture of SULPHATE OF MAGNESIA, CHLORIDE OF AMMONIUM, and AMMONIA; stir well, and set aside for an hour or two; **white crystalline precipitate** indicates *Phosphoric Acid.*

(*b*) **Arsenic is present.** Acidulate the *original* solution with HYDROCHLORIC ACID, saturate with HYDROSULPHURIC ACID gas, boil, and repeat the treatment with the gas, until the whole of the arsenic is precipitated; filter off the sulphide of arsenic, evaporate the filtered liquid to a small bulk, mix it with excess of ammonia, and test as in (*a*).

585*a*. *Silicic Acid.* Acidulate the original solution with HYDROCHLORIC ACID, evaporate to dryness, and digest the residue with dilute hydrochloric acid, white flakes remaining undissolved will consist of silica. (Confirm by the blowpipe (138, 138*a*). See note to (595).)

586. *Hydrocyanic Acid.* Mix the original solution with AMMONIA till it is slightly alkaline; add excess of yellow HYDROSULPHATE OF AMMONIA, boil, filter if necessary, evaporate till no more hydrosulphate of ammonia is perceptible to the smell, and add PERCHLORIDE OF IRON (if any black precipitate of sulphide is formed, filter it off); a **dark red color,** bleached by perchloride of mercury, indicates *Hydrocyanic Acid.*

587. *Oxalic*, *Tartaric*, and *Citric* Acids. Heat a portion of the ammoniacal solution (575) in a dish, till it is no longer alkaline, and add ACETATE OF LEAD as long as it causes any precipitate; collect the precipitate upon a filter, wash it once or twice with water, diffuse it through water, and decompose it by an excess of HYDROSULPHURIC ACID gas; filter off the sulphide of lead, evaporate the filtrate to a small bulk, and examine it as follows:—

(*a*) Test one part in a watch-glass, with CARBONATE OF POTASH added gradually, with constant stirring, so as to leave the liquid slightly acid; if any **crystalline precipitate** is formed, collect, and heat it, to see if it emits the peculiar odor of *Tartaric Acid* (435).

(*b*) Test another part in a somewhat dilute state, rendered alkaline by AMMONIA, with CHLORIDE OF CALCIUM; **white precipitate,** insoluble in ACETIC ACID, indicates *Oxalic Acid.*

(*c*) Test a third part for *Citric Acid* by neutralizing exactly with CARBONATE OF SODA, adding CHLORIDE OF CALCIUM, filtering off any precipitate, and boiling the clear solution; any precipitate which is formed must be collected on a filter, dried, and heated with STRONG SULPHURIC ACID to test for *Citric Acid* (444), by the **evolution of carbonic oxide,** burning with a blue flame on the approach of a light.

SECTION II.

THE COMPLEX SUBSTANCE IS KNOWN TO BE DISSOLVED IN AN ACID.[1]

Examination for Metals.

588. Reserve a small portion of the acid solution, and to the greater part add DILUTE HYDROCHLORIC ACID (unless that acid has been employed in preparing the solution).

(*a*) **No precipitate** is produced. Pass on to (589).

(*b*) **A precipitate** is produced. Collect it upon a filter, and reserve it for examination by (554). Proceed with the filtered solution, according to (589).

Hydrosulphuric Acid Test.

589. A few drops of the clear solution from (588) are

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[1] If the substance has been partly dissolved by nitric and partly by hydrochloric acid, the solution may be mixed and examined together if the precautions set down in the note on p. 201 be attended to.

mixed with three or four times their volume of a saturated solution of HYDROSULPHURIC ACID, and boiled.

(*a*) **No precipitate** except sulphur (see 492*b*) is produced. Treat the remainder of the solution (to which no hydrosulphuric acid has been added) according to (590).

(*b*) **A precipitate** which is not sulphur, is produced. The remainder of the clear solution from (588) is diluted with three or four times its bulk of water,[1] completely saturated with HYDROSULPHURIC ACID gas (see 547*b*), and filtered, the precipitate being reserved for examination by (556). The filtered liquid is mixed with more hydrosulphuric acid and boiled; if any precipitate is produced, it must be filtered off and examined with that first produced. The liquid should then be again diluted with water, saturated with hydrosulphuric acid, and boiled, these operations being repeated until the liquid gives no trace of precipitate or color when boiled with saturated hydrosulphuric acid. It is then further examined according to (590).

Hydrosulphate of Ammonia Test.

590. A small portion of the liquid in which hydrosulphuric acid gave no precipitate, or which has been filtered off from the precipitate, is tested with a few drops of hydrosulphuric acid, to be quite sure that nothing precipitable by that reagent is left, and is then mixed with CHLORIDE OF AMMONIUM,[2] AMMONIA in excess, and HYDROSULPHATE OF AMMONIA (disregarding any precipitate by ammonia alone), and gently heated.

(*a*) **No precipitate** is produced; proceed with the examination of the main portion of the solution by (591).

(*b*) **A precipitate** is produced; treat the whole of the solution in the same way, collect the precipitate upon a filter, and reserve it for examination by (594). Proceed to examine the filtered solution according to (591).

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[1] Should this produce any precipitate (antimony or bismuth), the solution should be heated, and hydrochloric acid added by degrees, until it becomes clear again.

[2] To prevent the precipitation of magnesia.

Carbonate of Ammonia Test.

591. A small portion of the solution in which hydrosulphate of ammonia gave no precipitate, or which has been filtered[1] off from the precipitate, is mixed with CHLORIDE OF AMMONIUM, AMMONIA in excess, and CARBONATE OF AMMONIA, and heated to boiling.

(*a*) **No precipitate** is produced; pass on to (592).

(*b*) **A precipitate** is produced; treat the whole of the solution in the same way, collect the precipitate upon a filter, and reserve it for examination by (567). Proceed to examine the filtered solution by (592).

Phosphate of Soda Test.

592. A small portion of the ammoniacal liquid in which carbonate of ammonia gave no precipitate, or which has been filtered off from the precipitate,[2] is mixed with PHOSPHATE OF SODA, well stirred with a glass rod (rubbed against the sides of the tube), and set aside for some minutes.

(*a*) **No precipitate** is produced; evaporate the main portion of the solution (to which no phosphate of soda has been added) to dryness in a small porcelain dish, and calcine the residue as long as any fumes of ammoniacal salts are evolved; examine the residue by (552).

(*b*) A **crystalline precipitate** is produced; presence of *Magnesium;* proceed with the main portion of the solution as in (551*b*) to examine for potassium and sodium.[3]

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[1] See note to (550).

[2] See note to (551).

[3] When a substance has been proved to be absolutely insoluble in water, it is unnecessary to examine it for potassium and sodium, unless antimonic, boracic, or silicic acid be present.

TEST FOR AMMONIUM.

593. Ammonium must be tested for by boiling the original solid substance in solution of POTASH (192), because the acid solution might possibly contain it as a product of the action of the acid upon some constituent of the original solid.

Examination of the Precipitate by Hydrosulphate of Ammonia.

594. This precipitate, when obtained from the acid solution of a complex substance, is liable to contain not only the metals of the third class, but, in addition, certain substances dissolved by the acid and reprecipitated merely in consequence of the neutralization of the acid by ammonia. The chief of these, which need be taken into account in the ordinary routine of analysis, are *Phosphates of Baryta*, *Strontia*, *Lime*, *Magnesia*, and *Alumina*, *Oxalates* of *Baryta*, *Strontia*, and *Lime*, and *Silica*.

595. The precipitate is well washed, upon the filter, till the washings leave no fixed residue when evaporated on a slip of glass, and dissolved in a mixture of three measures of DILUTE HYDROCHLORIC ACID with one measure of DILUTE NITRIC ACID, which may be heated and poured over the precipitate upon the filter. The solution is evaporated to dryness in a porcelain dish, and the residue is digested with a very little concentrated hydrochloric acid, until it is either entirely dissolved or a white flaky residue of *Silica* alone remains, which is filtered off,[1] washed, and examined before the blowpipe (138, 138*a*).

596. To the clear solution, add AMMONIA very carefully, drop by drop, until the precipitate formed at first redissolves with some difficulty on agitation; add ACETATE OF AMMONIA (prepared by mixing ammonia with acetic acid *in slight excess*) in considerable quantity.

(*a*) **A precipitate** is produced. Notice its appearance; **translucent** and **gelatinous,** *Phosphate of Alumina;* **opaque, gelatinous,** and **yellowish,** *Phosphate of Iron;* **pulverulent,** an *Oxalate.* Filter it off, and reserve it for examination by (601). Proceed with the filtered solution according to (597).

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[1] If the liquid passes through milky, titanic acid is probably present. It may be recognized by the pearly blue bead with microcosmic salt in the inner blowpipe flame.

(*b*) **No precipitate** is produced. Pass on to (597).

597. Dilute the solution with three or four volumes of water, and boil it for some minutes.

(*a*) **A precipitate** is produced. Collect it upon a filter, and reserve it for examination according to (603). Proceed with the filtered solution as in (598).

(*b*) **No precipitate** is produced. Pass on to (598).

598. To a small portion of this solution add a single drop of PERCHLORIDE OF IRON.

(*a*) **No precipitate** is produced. Examine the main portion of the solution by (600).

(*b*) **A precipitate** is produced, indicating the presence of *Phosphoric Acid.* Examine the main portion of the solution by (599).

599. Add PERCHLORIDE OF IRON to the solution until it has a pink or reddish color, and boil it for a few minutes. Filter off the precipitate, which contains the phosphoric acid in combination with iron, and proceed with the filtered liquid according to (600).

600. Evaporate the solution (which should have very little color if all the iron has been separated) to a small bulk, filter off any slight precipitate of peroxide of iron, mix the clear solution with an excess of AMMONIA, and saturate it with HYDROSULPHURIC ACID.

(*a*) **A precipitate** is produced. Filter it off, and reserve it for examination by (563*b*). Examine the filtered liquid as in (550, 551) for *Baryta, Strontia, Lime,* and *Magnesia,* which were contained in the original precipitate in the state of *Phosphates.*

(*b*) **No precipitate** is produced. Examine the solution according to (550, 551) for the *Alkaline*

Earths which were in combination with *Phosphoric Acid* in the original precipitate.

601. The **precipitate produced by acetate of ammonia in the cold** is well washed, and dissolved off the filter in hot DILUTE HYDROCHLORIC ACID, the solution mixed with excess of AMMONIA and HYDROSULPHATE OF AMMONIA. The solution is filtered off, and examined for *Phosphoric Acid* according to (376). The precipitate is treated as in (602).

602. This precipitate is washed, dissolved off the filter in hot DILUTE HYDROCHLORIC ACID, the solution boiled with a little nitric acid, till clear, evaporated to a small bulk, and mixed with a considerable quantity of ACETATE OF AMMONIA (prepared by mixing ammonia with acetic acid in slight excess).

(*a*) **No precipitate** is produced (but simply a red color. Add FERROCYANIDE OF POTASSIUM; **blue precipitate**; presence of *Iron*,[1] which has been contained in the precipitate of (596*a*) in the form of phosphate of peroxide of iron.

(*b*) **A precipitate** is produced. Filter it off, and test the solution for *Iron* as in (*a*). Wash the precipitate, dry it, and calcine it (with the filter if it be very small) in a crucible (to convert the oxalates of the alkaline earth into carbonates). Dissolve the calcined residue by heating with hydrochloric acid, filter the solution, if necessary, and add AMMONIA in excess. Filter off any precipitate of *Phosphate of Alumina*, and examine the filtered liquid for *Baryta*, *Strontia*, and *Lime* (originally present as *Oxalates*), by (591).[2]

603. The **precipitate produced in the first boiling with acetate of ammonia** is well washed, dissolved off the filter in hot DILUTE HYDROCHLORIC ACID, the solution mixed with excess of AMMONIA and HYDRO-

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[1] Test specially, in the original solution, to ascertain whether the iron existed there as protoxide or peroxide.

[2] If the filter has been incinerated, it must be remembered that it would contain a little lime and perhaps alumina. Incinerate a similar filter and examine the ash by the same process, for comparison.

SULPHATE OF AMMONIA, and heated to boiling. The solution is filtered off and tested for *Phosphoric Acid*, with a mixture of chloride of ammonium, ammonia, and sulphate of magnesia (376).

The precipitate produced by hydrosulphate of ammonia is washed, dissolved in hot DILUTE HYDROCHLORIC ACID, the solution boiled with a little NITRIC ACID, mixed with POTASH in excess, and boiled.

(*a*) **No precipitate** is produced. Test the solution for *Alumina* by acidulating with HYDROCHLORIC ACID and adding excess of AMMONIA (see note to 564*b*).

(*b*) **A precipitate** is produced. Filter it off, examine the solution for *Alumina* as in (*a*). Wash the precipitate, dissolve a small portion of it in HYDROCHLORIC ACID, and test with FERROCYANIDE OF POTASSIUM for *Iron* (see note to 602*a*). Dry the remainder, and test for *Chromium* by (222).[1]

Examination for Non-Metals and Acids in complex substances soluble in acids.[2]

A. *The analyst has a portion of the solid substance at his disposal.*

604. The experiments described in (467, 468, and 469), if carefully made, will establish the presence or absence of *Sulphurous*, *Hypochlorous*, *Acetic*, *Benzoic*, *Carbonic*, *Nitric*, *Hydriodic* (an iodide), and *Hydrofluoric* (a fluoride) Acids.

During the experiments to dissolve the substance (529), it will have been ascertained whether a sulphide is present or not.

605. Dissolve a portion of the substance by boiling with DILUTE HYDROCHLORIC ACID, filter the solution, if necessary, and examine by the following tests:—

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[1] The state of oxidation in which the chromium existed in the original solution (whether as oxide or as chromic acid) must be determined by special tests.

[2] Assistance may now be derived from the Table of Solubility in the Appendix. See note on page 212.

(*a*) CHLORIDE OF BARIUM; a **white precipitate** indicates *Sulphuric Acid.*

(*b*) AMMONIA in excess, and HYDROSULPHATE OF AMMONIA; heat the solution to boiling, and filter, if necessary; to the filtered liquid add a clear mixture of SULPHATE OF MAGNESIA, CHLORIDE OF AMMONIUM, and AMMONIA. Stir well with a glass rod, and set aside for some time. **Crystalline precipitate,** presence of *Phosphoric Acid.*[1]

(*c*) Dip a piece of TURMERIC PAPER into the solution, and dry it at a gentle heat; a brown **orange color** indicates *Boracic Acid.*

606. Boil a portion of the original substance with DILUTE NITRIC ACID, filter if necessary, and add NITRATE OF SILVER to test for *Hydrochloric Acid* (a chloride). See (584).

607. Boil a portion of the original substance with a strong solution of CARBONATE OF SODA for a few minutes; the bases will thus be converted into insoluble carbonates, and the acids will pass into solution.

(*a*) Acidify a portion of the solution very slightly with DILUTE HYDROCHLORIC ACID, dip a piece of TURMERIC PAPER into it, and dry it at a gentle heat, for *Boracic Acid.*

(*b*) Neutralize a portion of the alkaline solution very carefully with DILUTE NITRIC ACID, and add PERCHLORIDE OF IRON to detect *Benzoic*, *Hydroferrocyanic*, *Tannic*, *Gallic*, *and Acetic Acids.* See (580).

(*c*) Acidify a portion of the solution very slightly with DILUTE NITRIC ACID, heat it to expel free carbonic acid, render it very slightly alkaline with ammonia, and add CHLORIDE OF CALCIUM to detect *Oxalic*, *Tartaric*, and *Citric* Acids. See (576).

(*d*) Acidify a portion of the solution very slightly with DILUTE NITRIC ACID, and add NITRATE OF SILVER to detect *Hydriodic* and *Hydrocyanic* Acids. See (584).

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[1] If any arsenic acid be present, it must be separated as in (585*b*) before testing for phosphoric acid.

608. Boil a portion of the original substance with STRONG SULPHURIC ACID and COPPER-FILINGS to detect *Nitric Acid* (360).

B. *The analyst has only the acid solution at his disposal.*[1]

609. Dilute a portion of the solution with water, add DILUTE HYDROCHLORIC ACID, filter if necessary, and add CHLORIDE OF BARIUM; **white precipitate,** not dissolved by much water; *Sulphuric Acid.*

610. To another portion of the solution, add NITRATE OF SILVER.[2]

(*a*) **No precipitate** is produced. Pass on to (611).

(*b*) **A precipitate** is produced. Possible presence of *Hydrochloric Acid,*[3] *Hydriodic Acid,* and *Hydrocyanic Acid.* Examine according to (584).

611. Test for *Phosphoric Acid* in another portion of the solution according to (605*b*.)

612. Test for *Nitric Acid*[3] by (361).

613. To another portion of the original solution add a strong solution of CARBONATE OF SODA in excess, boil for a minute or two, filter if necessary, and neutralize the solution carefully with NITRIC ACID.

(*a*) Add PERCHLORIDE OF IRON to detect *Benzoic, Tannic, Gallic,* and *Acetic* acids. See (580).

(*b*) Acidify the carbonate of soda solution very slightly with DILUTE NITRIC ACID, heat it to expel free carbonic acid, render it very slightly alkaline with AMMONIA, and add CHLORIDE OF CALCIUM to detect *Boracic, Oxalic, Hydrofluoric, Tartaric,* and *Citric* acids. See (576).

614. Test specially for *Hydrocyanic Acid* by (429) in another portion of the original solution.

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[1] The presence of hydrosulphuric acid would be known by its odor.

[2] If hydrosulphuric acid be present, it should be expelled by a gentle heat before applying this test.

[3] Which may have been used to dissolve the original substance.

PART IV.

QUANTITATIVE ANALYSIS.

Introductory Remarks.

615. In the processes which I have now described, the object of the experimenter has been to ascertain what substances are present in a given salt or mixture of salts, which branch of analysis is called *qualitative.* I will now detail a few processes which have for their object the determination of the quantity of the ingredients of saline compounds: this branch of analysis is called *quantitative.* It is not my intention to enumerate the methods which have been devised for the separation and estimation of all, even of the more common compounds, but merely to give the student a general idea of the subject, by conducting him through a few simple examples of quantitative analysis, referring him, if he wishes for more extended information, to the larger works of Rose, Fresenius, and Parnell.[1]

616. I will first briefly describe some of the more important operations which have to be performed in the course of a quantitative analysis; and the student must bear in mind that the more care he bestows upon them, the more correct will be his results; as the loss of a single drop of liquid, or the presence of a very small quantity of soluble matter left in a precipitate, owing to carelessness in washing, will often occasion serious errors.

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[1] Traité Pratique d'Analyse Chimique, par H. Rose, of which an English translation by Dr. Normandy has recently appeared.

Chemical Analysis, Qualitative and Quantitative, by C. R. Fresenius, translated by Bullock.

Elements of Chemical Analysis by E. A. Parnell.

CHAPTER I.

OPERATIONS IN ANALYSIS.

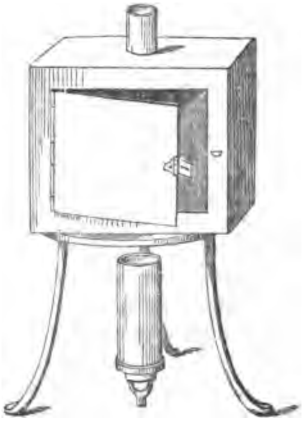
Pulverization.

617. Most substances may be reduced to sufficiently fine powder for analysis, by pounding in a common Wedgwood mortar; in some cases, however, it is first necessary to break the substance into small fragments, in one of iron or gun-metal; or in default of this, the substance may be loosely wrapped in strong brown paper, and struck with a hammer. When the substance is difficult of solution, as in the case of some siliceous minerals, it is sometimes necessary to reduce it to an impalpable powder in a small agate mortar; and on the fineness of this pulverization the success of an analysis often depends.

Drying.

618. Many substances, especially when in the state of powder, absorb moisture from the atmosphere, which, of course, adds to their weight. Before weighing out accurately the quantity of the substance for analysis, it is therefore necessary to deprive it of this hygroscopic moisture. This is generally done by heating it in a small basin on the water-bath or sand-bath, care being taken that the heat does not rise so high as to cause decomposition. The hot-water`box shown in Figure 72, is very convenient for drying substances at a low temperature: all the sides are made hollow, and filled with water,

Fig. 72.



Hot-water Drying Oven.

so that the temperature inside never rises higher than 212°. When a substance thus exposed ceases to lose weight, on being weighed at short intervals, it may be considered sufficiently dry. By using saline solutions, which boil at a higher temperature than water, a steady heat, considerably higher, may be obtained.[1]

Weighing.

619. Either 20, 25, or 33.3 grains will generally be found the most convenient quantity to take for quantitative experiments, regard being had to the number of constituents to be estimated, and the quantity of the substance at our disposal. The quantity may depend also on the method we intend to pursue, whether we propose to estimate all the ingredients from the same portion, or from two or more separate portions of the substance. If 20 grains are used, the results multiplied by five, will give the per-centage; or if 25 or 33.3 they must be multiplied by four or three. For most purposes the student will find a balance that is capable of weighing within one-tenth of a grain, sufficiently accurate; and it should be furnished with weights from one-tenth of a grain to 1000 grains.

620. A substance should never be weighed while warm, as it causes an upward current of air in its vicinity, which tends to buoy it up, and makes it appear to weigh lighter than it really is. In quantitative analysis, it is, of course, necessary to avoid the slightest loss in the weighed portion, as a deficiency in the weight of the ingredients would be the consequence, and the accuracy of the analysis seriously interfered with. Most substances in the state of fine powder, especially after having been recently ignited, are very prone to absorb moisture from the air; to obviate this, which would add materially to their weight, such substances should be weighed in a covered crucible, as soon as possible after cooling.

621. When, as is frequently the case, especially with liquids, a substance has to be weighed in a flask, dish, or other vessel, the latter may either be counterpoised with

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[1] Taylor's air-bath is now very generally used for drying substances.

strips of lead or shot, which are conveniently placed in a pill-box; or its weight may be previously noted, and afterwards deducted from the gross weight.

622. If the substance is weighed in a watch-glass, the last particles should be brushed off with a camel's hair pencil into the vessel to which the substance is transferred.

Solution.

623. Before the ingredients of a substance can be determined, either qualitatively or quantitatively, it is necessary to bring the substance into solution. For this purpose, water is to be preferred when the substance dissolves readily in it; and in the case of those compounds which are insoluble in water, one of the acids (generally hydrochloric) is employed, which has been found in the course of the preliminary examination, to be the best adapted for the purpose. (529 *et seq.*)

The dissolution of a substance is almost invariably assisted by heat, so that it is always advisable to use a vessel for the purpose which can be heated over a lamp without danger of fracture, as a small Berlin porcelain dish or glass flask. (Figs. 73

Fig. 73.

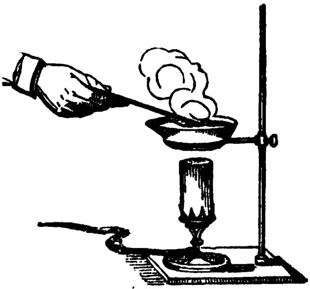


Fig. 74.

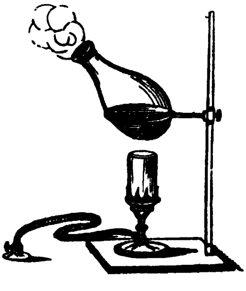
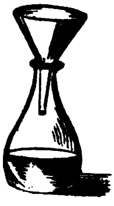


Fig. 75.

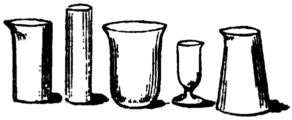


and 74.) The latter has the advantage of preventing loss by ebullition or spirting, as any particles of liquid that may be projected from the surface during ebullition, fall against the inner surface, and run back into the flask, especially if it is placed in an inclined position over the lamp. Occasional stirring facilitates the solution, and, as a general rule, the more finely the substance has been pounded, the more readily it dissolves. When a substance has to be digested in acid for a length of time, with the aid of heat, the evaporation of the acid may be in a great measure prevented, by placing a small glass funnel in the mouth of the flask (Fig. 75); the acid condenses, and runs back into the flask.

Precipitation.

624. When a substance is obtained in solution, the various compounds present are in most cases separated for the purpose of estimation, by adding to it some solution, which causes one or more of the ingredients to precipitate in the solid state; as, when we wish to estimate the quantity of sulphuric acid in any solution, we add to it a solution of chloride of barium, which, if added in sufficient quantity, causes the whole of the acid to precipitate in the form of sulphate of baryta, which being insoluble in water, may be washed without loss, and when dry is weighed; the weight of the sulphuric acid which it contains, may then be calculated from it.

Fig. 76.



Precipitating Glasses.

Precipitation is usually effected in upright glasses of the forms shown in Fig. 76. When precipitating a substance in quantitative analysis, it is important that sufficient of the precipitant be added to throw down *the whole* of the substance affected by it, as otherwise a deficiency in weight would be occasioned: this is easily ascertained by adding a drop of the precipitant to the solution filtered from the precipitate, which will cause a further precipitate if sufficient had not before been added. When the precipitate is at all soluble, as the bitartrate of potash, or ammonio-phosphate of magnesia, it is always advisable to allow the mixture to stand several hours before filtering, in order to insure the separation of the whole of the required salt. When the whole of the precipitate is thrown down, it is separated from the solution either by filtration or decantation.

Filtration.

625. The process of filtration is that most commonly adopted for separating a precipitate from the solution in which it was formed. The paper best adapted for the purpose is a thin white blotting paper, which should be free from visible holes, and should leave, when burnt, only a minute trace of inorganic matter.[1] Such a paper may be purchased from any of the dealers in chemical apparatus. It is convenient to keep a stock of filters ready cut, of a circular form, and of sizes varying from three to ten inches diameter. These may be made by having circular pieces of tin plate of the different sizes, and scoring round them with a pencil upon the paper, when several sheets may be cut through at once with scissors.

Fig. 77.

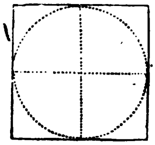
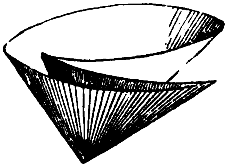


Fig. 78.



626. The filter, when required for use, is folded twice at right angles (Fig. 77), opened out into a conical form, and placed in a glass funnel, the sloping sides of which should open at an angle of about 60°, when it will be found to match the form of the folded filter, and will support it uniformly throughout. When placed in the funnel, the paper is moistened with water, for the purpose of causing the fibres to expand, and thus diminishing the

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[1] For many precipitates (sulphate of baryta, e. g.), the Swedish filtering paper is to be preferred to all other kinds.

size of the pores, without, at the same time, choking them with solid particles: if this is not done, and a solution mixed with a precipitate is poured into the dry filter, some of the finely divided particles of the precipitate are drawn into the pores by capillary attraction, and tend to prevent the passage of the clear solution through them. The filter should never be allowed to reach higher than the top of the funnel, as otherwise the weight of the liquid might cause the paper to give way; and there would also be danger of some of the solution running down the outside of the funnel, after passing through the projecting paper. It is even better that the funnel should project half an inch above the edge of the filter. When the filter is thus prepared, it may be supported either on the ring of a retort stand (Fig. 79), (for which the form shown at *a* is very convenient), or on a perforated block of wood placed on the glass intended to catch the filtered solution, the hole being made to fit the funnel, as shown in the section (Fig. 80).

Fig. 79.

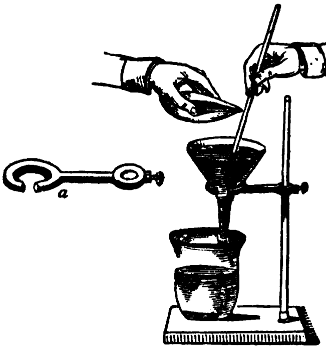
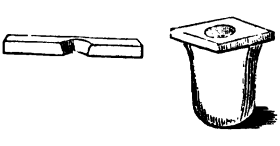


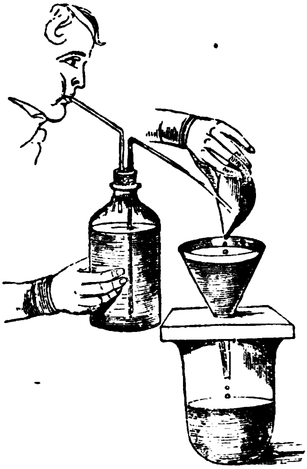
Fig. 80.



627. The solution to be filtered should be poured gently down a glass rod (Fig. 79), so as to fall on one of the slanting sides of the filter, and not into the apex, as that would endanger the

bursting of the paper, and cause splashing. A liquid, holding a precipitate in suspension, should never be poured higher than within half an inch of the margin of the filter. When the whole of the mixture has been poured on the filter,[1] fresh water should not be added for the purpose of washing, until the whole of the solution has passed through; then by means of a washing-bottle (89), the precipitate left on the filter is well washed; the current of water being applied first towards the upper part of the filter, and directed gradually downwards (Fig. 81). When the filter has been thus nearly filled up with water, allow the whole to run through before adding any more, and then repeat the washing until a drop of the filtered liquid leaves no fixed residue when evaporated on a piece of glass. If the precipitate, while standing in the filter, cakes together into lumps, these must be broken up by directing upon them a strong current of water from

Fig. 81.



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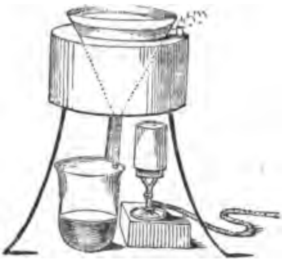
[1] In removing the last particles of the precipitate from the vessel in which it has been deposited, a glass rod, with one end covered with a cap of caoutchouc (160), will be found very convenient; in the case of a flask, the rod may be bent, or a tuft at the end of a quill may be used.

the washing-bottle, as otherwise the water would not penetrate them, and some of the soluble matter would escape removal.[1]

628. It is sometimes necessary to keep the mixture hot during filtration, to prevent any of the soluble ingredients solidifying: this may be done very conveniently, by placing the funnel in a zinc or copper box of the form shown in Fig. 82, which may be kept full of hot water, and boiling, if necessary, over a lamp.

Fig. 82.



Hot-water Filtering Stand.

629. The liquid is generally filtered into a beaker glass, and occasionally into flasks or dishes; it is always advisable to cause the stream to run gently down the side of the vessel, and not to fall drop by drop into the centre of the glass, as this would cause splashing and probably some loss. It occasionally happens that some of the precipitate passes through with the filtered solution, as may be seen in the case of freshly precipitated oxalate of lime or sulphate of baryta. When this takes place, it is sometimes necessary to pass it through the filter twice or three times before it comes through quite clear. This may, however, in most cases, be obviated by boiling the mixture before filtering, which causes the finely divided particles of the precipitate to aggregate together. The presence of some saline matters in solution, also, sometimes prevents a precipitate passing through: chloride of ammonium, for example, exerts this property with sulphate of baryta.

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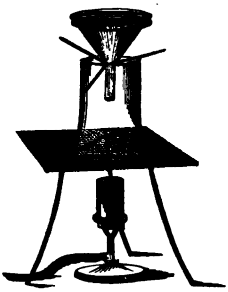
[1] Many precipitates are washed with hot water; for this purpose a flask must be employed which is furnished with tubes like the washing-bottle, and can be heated over a lamp. In many cases, for example, in that of oxide of copper precipitated by potash, it will be found advantageous to allow the precipitate to subside, decanting the clear liquid by degrees through the filter, and to boil the precipitate with water, afterwards allowing it to subside as before. When this process has been repeated once or twice, the precipitate may be thrown upon the filter, and the washing completed.

630. When the precipitate on the filter is completely washed, the funnel, with its contents, is placed on a small tripod, or retort stand, on the warm sand-bath, or near a fire, when the precipitate will gradually dry; it may then be separated from the filter, ignited in a small platinum or porcelain crucible (639), (unless decomposable at a high temperature), and weighed.

631. Precipitates may be very rapidly dried by supporting the funnel and filter containing them in a beaker with the bottom out, and placing this upon a piece of strong coarse wire network, resting upon a tripod stand (Fig. 83), over an Argand burner with a very small flame. In order to prevent any dust from falling into the precipitate, it is advisable to cover the funnel with a piece of filter-paper, secured by folding down over the rim.

Fig. 83.

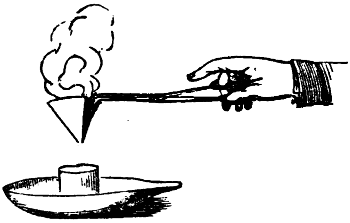


632. In cases where the quantity of the precipitate is very small, and where it will not bear a red heat without decomposition, it may be weighed in the filter, which should be placed in a covered porcelain crucible of known weight, dried at 212°, and weighed before the precipitate is collected upon it. When the precipitate on the filter has been thoroughly washed, the latter is partly dried in the funnel, placed in the crucible, dried as before, and as soon as it is cold, again weighed; when the increase in weight will, of course, be that of the precipitate.

633. It is often necessary, before weighing a precipitate, to burn the filter containing it. After the greater part of the precipitate has been removed, the filter is held with a pair of pliers, and set fire to, over the platinum crucible in which the precipitate is to be ignited, the crucible being placed in a basin, or upon a sheet of glazed paper, in case any of the ashes should fall over its sides (Fig. 84); these are then collected and ignited in the crucible (639), together with the portion of the precipitate previously removed from the filter, until the whole of

the charcoal derived from the paper is burnt away. In cases of great accuracy, the weight of the paper ashes, ascertained by weighing those derived from six or eight similar filters, must be deducted from the gross weight; when the paper is good, however, it does not contain more than one to three-thousandths of its weight of inorganic matter, so that this precaution is scarcely necessary in ordinary cases of analysis.

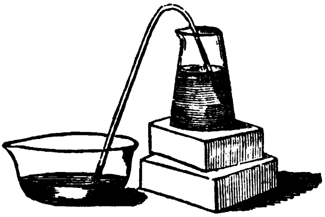
Fig. 84.



Decantation.

634. When a precipitate is found to subside rapidly to the bottom of the liquid, and when it is known to be very insoluble in water, it may be washed by DECANTATION, instead of on a filter, and in many cases this is the more expeditious method. The mixture is placed in an upright jar or beaker, which is then filled up with water, and allowed to stand until the precipitate has subsided to the bottom, leaving the superincumbent liquid clear.

Fig. 85.

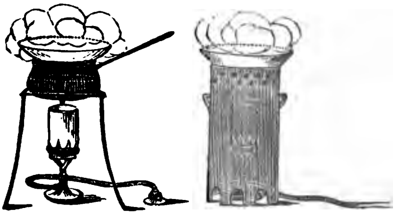


The latter is then removed with a syphon (Fig. 85), or carefully poured off, and the jar again filled up with distilled water, the process being repeated until all the soluble matter has been removed. The wet precipitate is then placed upon a filter, or dried in a dish, and weighed.

Evaporation.

635. The process of evaporation is generally most conveniently effected in Berlin porcelain evaporating basins, either on a sand-bath or over a lamp. Care must be taken, in quantitative experiments, that no loss is occasioned by spirting, and, on this account, it is safer not to allow the liquid absolutely to boil. When a saline solution has to be evaporated to dryness, it often becomes covered, when concentrated, with a pellicle of solid matter, preventing the escape of the steam, which, being thus confined, occasionally causes some of the mixture to be projected violently from the basin. The best way of avoiding this, is to stir the mixture constantly with a glass rod, from the time when the pellicle begins to form, until it is evaporated to dryness.

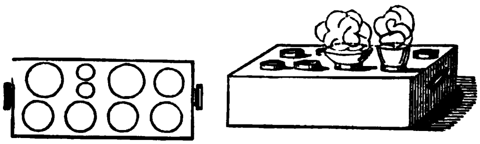
Fig. 86.



636. It is often advisable, and in the case of many liquids, as those containing organic matter, necessary, to evaporate over a water-bath; by this means the heat is never allowed to rise higher than 212°. For this purpose, a common saucepan, or almost any vessel used for boiling water in, may be employed, placing the dish containing the solution over the top, as shown in Fig. 86, so as to expose it to the action of the steam. For the laboratory, a convenient form of water-bath is shown in Fig.

87; it may be made of copper or zinc plate, and the holes should be fitted with lids, to cover them when not wanted.

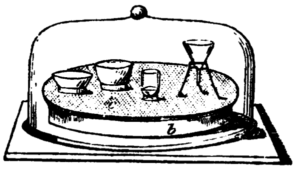
Fig. 87.



Hot-water Bath.

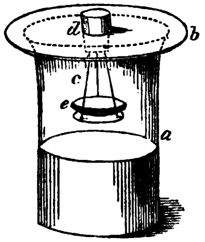
637. A convenient method of drying certain substances which are liable to decomposition at a slightly elevated temperature, is to place them under the receiver of the air-pump (Fig. 88), over an open pan of strong sulphuric acid; the latter absorbs the moisture which rises from the substance, and a gradual and complete desiccation may be effected at ordinary temperatures. The dishes containing the substance to be dried may be placed on a sheet of perforated zinc, resting on the pan of acid. If an air-pump is not at hand, the same effect may be produced, though more slowly, by placing the receiver inclosing the substance and acid, upon a flat piece of glass, or the temporary arrangement shown in Fig. 89 may be adopted, *a* being a beaker containing sulphuric acid, over which the substance to be dried is suspended in the dish *e*. Owing to the slowness of the evaporation, this

Fig. 88.



Desiccation over Sulphuric Acid.

Fig. 89.



Desiccation over Sulphuric Acid.

method is well adapted for obtaining large and well defined crystals from saline solutions, &c.

638. When a uniform temperature is required, higher than 212°, it may be obtained by immersing the dish or flask containing the substance to be evaporated, in a bath of oil or some saline solution, the boiling point of which is near the desired temperature. Olive oil may be heated to nearly 500° without suffering much decomposition, and forms an extremely useful bath for many purposes, since, by regulating the lamp, and placing a thermometer in the oil, any lower temperature can readily be kept up. The following list shows the boiling points of a few saturated saline solutions, which will occasionally be found useful for this purpose.

A saturated solution (at 60°) of

Bitartrate of potash	boils at 214°
Sulphate of copper	" 216°
Chlorate of potash	" 218°
Carbonate of soda	" 220°
Alum	" 220°
Borax	" 222°
Chloride of sodium	" 224°
Chloride of calcium	" 230°
Tartrate of potash	" 234°
Chloride of ammonium	" 236°
Nitrate of potash	" 238°
Rochelle salt (tartrate of potash and soda)	" 240°
Nitrate of soda	" 246°
Acetate of soda.	" 250°

By adding a further quantity of most of these salts to the *hot* solutions, and thus making them more concentrated, considerably higher temperatures may be obtained.

Ignition.

639. It is generally necessary, previous to weighing a precipitate, in quantitative analysis, to heat it to redness,

Fig. 90.

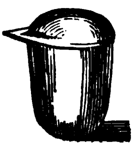
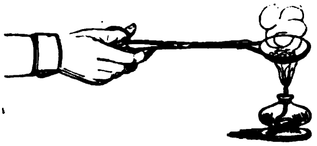


Fig. 91.



in order to insure perfect dryness. This is usually done in a weighed platinum or porcelain crucible, either in a furnace or over a lamp. When the crucible is to be heated in the furnace or open fire, it should be inclosed in one of earthenware, to protect it from contact with the coals and dirt, a little magnesia being interposed between the two (note to 480). If the lid is made of the form shown in Figure 90, it may also be used as a capsule, independently of the crucible (Fig. 91).

640. When gas is available, scarcely any other source of heat is necessary for the purpose. A small platinum crucible may be heated to low redness over the naked flame, resting on a small wire triangle placed on the top of the chimney (Fig. 92).

A mixture of gas and air, however, gives a much more intense heat, owing to the more perfect oxidation of the combustible matter; such a mixture is easily obtained by placing a small piece of wire gauze over the chimney, and applying a light to the mixture as it rises through the gauze[1] (Fig. 93). The crucible may be supported on a wire triangle, or in a *jacket* of thin iron plate, *a* (Fig. 93). In this simple manner, a very powerful lamp furnace is easily obtained, which may be varied in size and form to suit the purposes for which it may be required. Thus, Mr. Hardwich and Mr. Beale have succeeded in

Fig. 92.

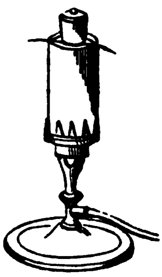
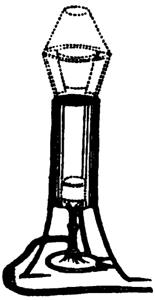
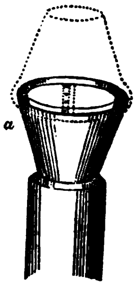


Fig. 93.



Crucible Jacket.

Fig. 94.



Crucible Jacket.

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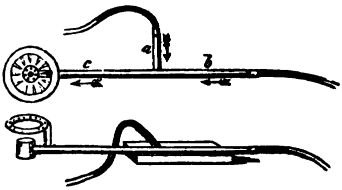
[1] Bunsen's burner acts upon the same principle, and is even more efficient.

making a furnace admirably adapted for heating tubes of a considerable length. An ingenious arrangement is made for gradually increasing the length of the flame along the tube; so that for such purposes as the ultimate analysis of organic substances, it may be substituted, with great advantage, for Liebig's charcoal furnace.[1]

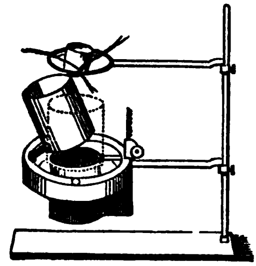
Fig. 95.



Solly's Gas Furnace.

641. Mr. Solly also has contrived a very convenient form of lamp, in which he mixes air with the gas by means of a pair of double bellows, forming a series of blow-pipe jets, the combined action of which is very powerful, and is capable of keeping a large platinum crucible almost white hot for any length of time. The gas pipe *a* (Fig. 95) joins the tube *b*, bringing air from the bellows placed underneath, forming in *c* an inflammable mixture: this burns as it issues from the apertures in the burners, the number of which may be multiplied to almost any extent. The crucible may stand on a wire triangle resting on the circle, and the whole may be surrounded by a jacket of thin iron plate, to prevent loss of heat by radiation.[2]

Fig. 96.



Rose's Spirit Lamp.

642. When gas cannot be had, the best lamp for heating a small platinum crucible to redness, is that known as Rose's, the form of which is shown in Figure 96; either alcohol or pyroxylic spirit may be burnt in it.[3]

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[1] The results obtained with this gas-furnace as improved by Dr. Hofmann are far more satisfactory.

[2] It is particularly advantageous to have a concave piece of tin-plate, about six inches in diameter, fixed beneath the burner to check the ascending column of cold air.

[3] The Berzelius spirit-lamp is safer than Rose's, since the reservoir of spirit is not placed so near the flame.

Calculation of Results.

643. When the weight of a precipitate has been ascertained, it is necessary to calculate that of the constituent, the weight of which we wish to learn, and this is readily done according to the well-known laws of combination in definite proportions.

For example, let us suppose that we have to determine the per-centage of sulphuric acid (SO_3) in dry sulphate of soda (NaO,SO_3): we dissolve twenty grains of the salt in water, precipitate the sulphuric acid by means of chloride of barium, and weigh the sulphate of baryta thus obtained: from this we have to deduce the weight of the sulphuric acid which it contains; and lastly, to calculate from this the per-centage equivalent to it. We find the weight of the sulphate of baryta obtained to be 32.80. Knowing the atomic weight of sulphate of baryta (BaO,SO_3) to be 116.5, and that of sulphuric acid (SO_3) to be 40, it is easy to calculate how much of the acid is contained in 32.80 grains of the precipitate, thus:—

$$\overbrace{116.5}^{\substack{\text{Atc. wt. of sulph. of}\\ \text{baryta.}}} \;:\; \overbrace{40}^{\substack{\text{Atc. wt. of sulph.}\\ \text{acid.}}} \;::\; \overbrace{32.80}^{\substack{\text{Wt. of sulph. of}\\ \text{baryta obtained.}}} \;:\; \overset{\substack{\text{Wt. of sulph. acid}\\ \text{in 32.80 grs. of}\\ \text{sulphate of baryta.}}}{x=11.26.}$$

Thus we find that twenty grains of the dry sulphate of soda contain 11.26 of sulphuric acid; and we have now only to reduce it to a per-centage, to complete the calculation, thus:—

$20 : 11.26 :: 100 : x = 56.30$ sulphuric acid in 100 parts of dry sulphate of soda. Or, as 20 is the fifth part of 100, the same result may be obtained by simply multiplying by 5. $11.26 \times 5 = 56.30$.

CHAPTER II.

EXAMPLES IN QUANTITATIVE ANALYSIS.

644. In the following examples it is assumed that the nature of the substances has been already ascertained by a qualitative examination; since it is always necessary, before proceeding to estimate the constituents of a compound, that we should know what those constituents are.

SECTION I.

Quantitative Analysis of Sulphate of Copper.
$(CuO,SO_3,HO+4Aq.)$[1]

Estimate the quantity of oxide of copper (CuO), sulphuric acid (SO_3), and water (HO), in sulphate of copper.

(1.) *Estimation of the Oxide of Copper.*

645. Dissolve twenty grains of the salt in eight or ten ounces of water, in a beaker, and gently boil the solution upon a sand-bath. Add to it, while boiling, a solution of potash in considerable excess, which will throw down the black oxide of copper.

$$CuO,SO_3+KO=\mathrm{CuO}+KO,SO_3.$$

The mixture is poured upon a filter (see note to 627), and carefully washed with boiling water, until the whole of the soluble matter is removed (627). The precipitate in the filter is then dried, separated from the filter, ignited, with the precautions given at (633), in a porcelain crucible, and weighed.[2]

When the precipitate consists, as in this case, of the substance the weight of which we wish to ascertain, uncombined with other matter, we have only to reduce the amount thus obtained, to a percentage, thus:—

20 : weight of the precipitate of oxide : : 100 : x = percentage of oxide of copper. Or, in other words, multiply the weight by 5.

(2.) *Estimation of the Sulphuric Acid.*

646. The acid may be estimated either from the same portion of the substance as was used in determining the oxide (in which case the solution filtered from the preci-

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[1] The per-centage composition calculated from this formula is—

Oxide of copper	31.9
Sulphuric acid	32.1
Water of crystallization	28.8
Water of constitution	7.2

[2] Since oxide of copper is very hygroscopic, the crucible and its contents should be weighed immediately after cooling; it is even more satisfactory to allow the cooling to proceed under a glass shade containing a vessel of oil of vitriol.

pitated oxide must be concentrated by evaporation (635)), or a fresh portion of the salt may be used. The latter method is in this case the simplest.

647. Dissolve twenty grains of the sulphate in water as before, acidify it with a few drops of hydrochloric acid, and add a solution of chloride of barium (*BaCl*) as long as it causes any precipitate. Sulphate of baryta (BaO,SO_3) is thus thrown down, and the whole of the sulphuric acid is in this way removed from the solution. As the mixture in its present state would not filter well, it is advisable to boil it before filtering, and to allow the precipitate to subside; when the supernatant liquid is quite clear, it may be carefully poured through the filter (of Swedish paper) without disturbing the precipitate, over which some boiling water is then poured; when the precipitate has again subsided, the water is decanted through the filter, and replaced by a fresh portion. A very small beaker is then placed beneath the funnel, in case any of the precipitate should pass through the filter, and the precipitate is carefully rinsed upon the filter, which should never be filled to more than one-fourth of its capacity. The precipitate is well washed with hot water on the filter, dried, ignited in a platinum or porcelain crucible, and weighed. Then, knowing the atomic weight of sulphate of baryta to be 116.5, and that of sulphuric acid (SO_3) to be 40, or, in other words, that every 116.5 parts of the former contain 40 of the latter, it is easy to calculate the quantity of sulphuric acid contained in the precipitate, thus:—

$$116.5 : 40 : : \text{weight of the precipitate} : x = \left\{\begin{array}{l}\text{The sulphuric acid in}\\ \text{20 grains of sulphate}\\ \text{of copper.}\end{array}\right\}$$

which number, multiplied by five, will represent the percentage of sulphuric acid in crystallized sulphate of copper.

(3.) *Estimation of the Water of Crystallization.*

648. Twenty grains of the finely powdered salt are heated in a counterpoised crucible placed in a water-oven (618) until it ceases to lose weight. The loss is multiplied by five to obtain the percentage.

(4). *Estimation of the Water of Constitution.*

649. The salt dried in the last experiment is heated on the sand-bath or in an air-bath to about 300°, until its weight is constant.[1]

SECTION II.

Quantitative Analysis of Chloride of Sodium ($NaCl$).[2]

650. Reduce the pure salt to powder and calcine it in a porcelain or platinum crucible to expel moisture.

(1.) *Estimation of the Sodium.*

651. Dissolve twenty grains of the salt in half an ounce of water in a large porcelain crucible, previously weighed, with its cover. Mix the solution carefully, with a glass rod, with about twenty drops of strong sulphuric acid, and evaporate it very carefully (635) to dryness over an argand burner, the heat being gradually increased towards the last, as long as any dense white fumes of sulphuric acid are perceptible. The crucible is then covered, and the residue strongly calcined for a few minutes; after cooling, a small fragment of carbonate of ammonia is introduced (to decompose any traces of bisulphate of soda which might be left), and the calcination repeated. The weight of the residue (sulphate of soda) is then determined, and that of the sodium is calculated from it:—

$$\underbrace{\text{At. wt. of sulph. soda.}}_{71} : \underbrace{\text{At. wt. of sodium.}}_{23} :: \underbrace{\text{Wt. of sulph. soda obtained.}}_{a} : \underbrace{\text{Sodium in 20 grs. of salt.}}_{x}$$

which, when multiplied by five, gives the per-centage of sodium.

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[1] When the quantity of water has to be estimated in salts which cannot bear the necessary heat without the volatilization of a portion of their acid, the salt should be intimately mixed with five or six times its weight of protoxide of lead, or some other strong base, before exposure to heat: this combines with, and fixes, any of the acid that may be disengaged from the other base.

[2] The percentage composition is—

Sodium	39.32.
Chlorine	60.68.

(2.) *Estimation of the Chlorine.*

652. Twenty grains of the chloride are dissolved in three or four ounces of water, in a beaker; the solution is then heated, acidified with a few drops of nitric acid, and treated with a solution of nitrate of silver, as long as it causes any precipitate.

$$NaCl + AgO,NO_5 = \mathrm{AgCl} + NaO,NO_5.$$

The mixture is then briskly stirred with a glass rod, boiled for a few minutes (as otherwise a portion of the precipitate would pass through the pores of the paper (629)), and filtered. The precipitated chloride of silver is thoroughly washed with distilled water on the filter, and dried; it is then removed from the paper, and gently ignited in a counterpoised porcelain crucible, until it fuses into a waxy mass, and weighed.[1]

653. The atomic weight of chloride of silver is 143.5, and that of chlorine 35.5, so that we deduce the weight of the chlorine from that of the chloride, thus:—

$$14.35 : 35.5 :: \text{wt. of the chloride of silver} : \left\{ \begin{array}{l} \text{Wt. of chlorine contained in 20} \\ \text{grains of chloride of sodium.} \end{array} \right\}$$

Then, for the per-centage, multiply by five.

SECTION III.

Quantitative Analysis of Tartar Emetic (tartrate of Antimony and potash, $KO,SbO_3, C_8H_4O_{10} + Aq$).[2]

(1.) *Estimation of the Water.*

654. The water is estimated in the manner described at (648).

---

[1] The filter, with the adhering precipitate, should be incinerated as directed at (633), the ash being added to the fused chloride in the crucible.

[2] The per-centage composition is—

Potash	14.07.
Oxide of Antimony	43.71.
Tartaric Acid	39.52.
Water	2.70.

(2.) *Estimation of the Oxide of Antimony.*

655. Dissolve twenty grains of the salt in four ounces of water, in a beaker, add hydrochloric acid by degrees till the precipitate at first produced is redissolved, and transmit a current of hydrosulphuric acid gas (687) until the solution smells very strongly of the gas, even after having been removed from the apparatus, and stirred for some time.[1] The orange precipitate of tersulphide of antimony (SbS_3) is collected upon a filter which has been dried in the water-oven and weighed (632); the precipitate is washed until a drop of the washings no longer leaves any considerable residue when evaporated upon a slip of glass (the washings, together with the filtrate, being carefully preserved), and dried in a current of warm air (631), the filter being afterwards removed from the funnel and dried in the water-oven (632) until its weight no longer decreases, after half an hour's drying. By deducting the weight of the dry filter, that of the sulphide of antimony is ascertained, from which the per-centage of antimony is thus calculated:—

$$\underbrace{\text{At. wt. of sulphide of antimony.}}_{170} : \underbrace{\text{At. wt. of ox. of antimony.}}_{146} :: \underbrace{\text{Wt. of sulphide of antimony obtained.}}_{a} : \underbrace{\text{Ox. of antimony in 20 grs. tartar-emetic.}}_{x}$$

which, when multiplied by five, gives the per-centage of oxide of antimony.

(3.) *Estimation of the Potash.*

656. Test the filtrate and washings from the sulphide of antimony with hydrosulphuric acid to prove that all the antimony has been precipitated, and evaporate them carefully (635) in a dish to about an ounce; transfer this down a glass rod to a large porcelain crucible (carefully rinsing the dish with water), and evaporate to dryness upon the water-bath (636). Place the crucible containing the dry residue over an Argand burner, loosely cover it, and apply a gradually increasing heat until the whole of

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[1] The delivery-tube of the apparatus should be removed with the beaker, to avoid the loss of any adhering sulphide of antimony. Any particles of the precipitate may be removed from the inside of the tube by means of a tuft at the end of a quill pen.

the tartaric acid is decomposed. Open the crucible and continue to heat the residue to dull redness until as much of the carbon as possible has been burnt off. Dissolve the residue in about half an ounce of hot water, acidulate the solution cautiously with hydrochloric acid, filter from any undissolved carbon, and wash the filter with water until a drop of the washings leaves no considerable residue when evaporated on a glass plate; mix the filtrate and washings with excess of bichloride of platinum, and evaporate the mixture to dryness on a water-bath (636). Treat the residue with alcohol, to dissolve out the excess of the bichloride, and wash the insoluble double chloride of platinum and potassium (186) with fresh alcohol on a weighed filter (632). If it is found that the alcohol which is added to the dried residue does not become colored strongly yellow, it is owing to there being no excess of bichloride of platinum present. In this case, a little more bichloride of platinum should be added, and the mixture again evaporated as before, and subsequently treated with alcohol. The precipitate is washed with alcohol on the filter till the washings pass through colorless, dried in the water-oven, and weighed.

657. The atomic weight of the double chloride ($KCl, PtCl_2$) being 244.1, and that of potash being 47, we deduce the quantity of potash contained in the twenty grains of tartar emetic, thus:—

$$244.1 : 47 :: \left\{\begin{array}{c}\text{Wt. of double chloride of}\\ \text{platinum and potassium.}\end{array}\right\} : \left\{\begin{array}{c}\text{Wt. of potash contained in}\\ \text{20 grs. of tartar emetic.}\end{array}\right\}$$

the per-centage being obtained by multiplying the result by five.

SECTION IV.

Quantitative Analysis of Magnesian Limestone; consisting of Carbonate of Lime (CaO,CO_2), *Carbonate of Magnesia* (MgO,CO_2), *Carbonate of Oxide of Iron* (FeO,CO_2), *Silica, Alumina and moisture.*

Determine the quantity of lime, magnesia, alumina, oxide of iron, carbonic acid, silica, and moisture. Reduce about 100 grains of the mineral to moderately fine powder.

(1.) *Estimation of the Hygroscopic Moisture.*

658. Weigh fifty grains of the powdered mineral in a small counterpoised crucible or evaporating dish, and dry it in the air-bath, or on the hot part of the sand-bath; weigh it at intervals of a quarter or half an hour until it ceases to lose weight (620). The loss will be the quantity of moisture in fifty grains, which, when multiplied by two, will give the per-centage.

(2.) *Estimation of the Sand.*

659. Weigh twenty-five grains of the powdered mineral in a counterpoised flask, moisten it with a little water, and add dilute hydrochloric acid in small quantities, to avoid too violent effervescence (623). When the greater part is dissolved, warm it with a little fresh acid, which will dissolve everything but the small quantity of sand, and possibly a little clay. The mixture is now filtered, and the precipitate thoroughly washed, the solution being retained for subsequent examination (660); the filter containing the precipitate is then ignited and weighed. The weight of the siliceous residue, multiplied by four ($25 \times 4 = 100$), gives the per-centage of sand in the stone.

(3.) *Estimation of the Silica.*

660. The acid solution filtered from the siliceous residue is mixed with a little nitric acid, evaporated to dryness, and the residue digested with hydrochloric acid till nothing but white flakes of silica is left undissolved; the silica is collected upon a filter, washed till the washings are no longer acid, dried, very carefully ignited[1] (silica being very apt when heated to be carried away by a current of air), and weighed.

(4.) *Estimate of the Oxide of Iron and Alumina.*

661. The acid solution filtered from the silica is mixed with chloride of ammonium and an excess of ammonia,

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[1] In igniting silica it is advisable to remove it as completely as possible from the filter and to ignite it in a covered crucible, the filter being incinerated separately and the ash added to the ignited silica after cooling.

and gently heated; the precipitate of alumina and sesquioxide of iron is collected upon a filter, well washed, dried, ignited and weighed.[1]

(5.) *Estimation of the Lime.*

662. The solution filtered from the alumina and oxide of iron is now to be mixed with oxalate of ammonia as long as it causes any precipitate. This throws down the lime as oxalate, while the magnesia remains in solution, not being precipitated by oxalate of ammonia in the presence of chloride of ammonium, which is contained in the solution. The mixture is boiled (629), and filtered, and the precipitate washed and dried; the solution being retained for subsequent examination (663). The oxalate of lime is now removed from the filter and ignited (633), by which means it is decomposed, and converted into carbonate of lime; at a red heat, however, a portion of the newly-formed carbonate is decomposed, the carbonic acid being expelled, and thus leaving a little caustic lime (CaO) mixed with the carbonate. When cool, it is sprinkled with powdered carbonate of ammonia and a little water, and again gently heated, to expel the excess of carbonate of ammonia; the whole of the lime is thus converted into carbonate, in which state it is weighed.

The atomic weight of carbonate of lime being 50, and that of lime 28, the weight of the latter is thus calculated:—

$$50 : 28 :: \text{wt. of carbonate obtained} : \left\{ \begin{matrix} \text{Wt. of lime contained in 25} \\ \text{grains of the mineral.} \end{matrix} \right\}$$

which number, multiplied by four, gives the per-centage of lime.

(6.) *Estimation of the Magnesia.*

663. The solution filtered from the oxalate of lime is now to be concentrated by evaporation, and treated with

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[1] The circumstance that the iron is contained in this precipitate as sesquioxide, though the limestone contains it in the form of protoxide, introduces a slight inaccuracy into the analysis ($2FeO : Fe_2O_3 :: 72 : 80$), but it is seldom necessary to separate the sesquioxide of iron from the alumina.

a mixture of phosphate of soda and a considerable excess of caustic ammonia; when the mixture is set aside for a few hours, being stirred at intervals with a glass rod. The magnesia is thus thrown down as the double phosphate of ammonia and magnesia (212). Before filtering, a little of the mixture should be tested with a few drops more phosphate of soda, and allowed to stand a short time; when, if no fresh precipitate is formed, it may be concluded that a sufficient quantity of the precipitant has been added.

The mixture is now filtered, washing always with water containing a little free ammonia, as the double phosphate is slightly soluble in pure water (any of the precipitate that adheres to the sides of the glass being separated by means of a caoutchouc cap, which must be well washed from all adhering particles), and the precipitate, after drying, is ignited in a small counterpoised platinum or porcelain crucible, and weighed. During ignition, the double phosphate is decomposed into phosphate of magnesia ($2MgO,PO_5$), the water and ammonia being driven off (212).

The atomic weight of the phosphate of magnesia thus formed being 111.4, and that of magnesia 20.2, the quantity of magnesia contained in the precipitate is thus calculated:—

111.4 : 40.4[1] : : wt. of phosphate : wt. of magnesia in 25 grs. of the mineral.

which, when multiplied by four, gives the per-centage of magnesia in the mineral.

(7.) *Estimation of the Carbonic Acid.*

664. The carbonic acid is estimated in the following manner:—

Take a light piece of tube, *b* (Fig. 97), three or four inches long, with one end drawn out so as to leave only a small opening, and fill it with fragments of chloride of calcium, putting in a loose plug of tow or cotton wool at each end, in the manner already described (159). Bend a piece of quill tubing in the form shown at *d*

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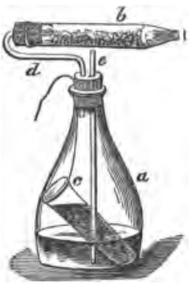
[1] $40.4 = 20.2 \times 2$; because each equivalent of the phosphate ($2MgO, PO_5$) contains two equivalents of magnesia.

Fig. 97.



Carbonic Acid Apparatus.

(86), and by means of perforated corks, connect the two tubes with a light flat-bottomed flask, capable of holding six or eight ounces of water. Make another perforation in the cork of the flask to receive a very small tube *e*, passing down nearly to the bottom of the flask, and projecting about two inches above the cork; the upper orifice of this tube is stopped with a small plug of bees-wax. Select a small test-tube, about an inch and a half long, and having counterpoised if accurately, introduce into it twenty grains of the powdered limestone.

Pour into the flask six drachms of strong hydrochloric acid and six drachms of water; attach the tube *c* to a horsehair, and sling it in the flask, securing the hair between the cork and the glass, so that the tube containing the marble may hang safely above the surface of the acid, while the whole apparatus is being accurately weighed. Now gradually incline the flask, so as to allow the acid to act upon the carbonate; the carbonic acid is disengaged with effervescence; is deprived, while passing over the chloride of calcium, of the moisture with which it is impregnated; and passes off through the small aperture in the tube *b*, leaving the apparatus of course lighter than before.

$$\mathrm{CaO,CO_2} + HCl = CaCl + HO + \mathrm{CO_2}.$$

665. When the effervescence has ceased, the flask should be gently warmed, and when cool again, the wax plug may be removed from the tube *e* (being of course saved to be weighed with the apparatus), and air drawn through the flask by applying the lips to the tube *b*, to abstract the whole of the carbonic acid with which it is filled; and which, being heavier than common air, would add to its apparent weight. The whole apparatus is then again weighed; the loss of weight being of course that of the carbonic acid expelled. By multiplying this loss by 5 (20×5=100), the percentage of carbonic acid in the limestone is obtained.

666. Thus we shall have determined the percentage of the—

Water . . . .
Silica . . . .
Alumina }
Peroxide of Iron } . . . . .
Lime . . . .
Magnesia . . . .
Carbonic Acid . . . . . .
Sand }
Clay } . . . .
100.00

which, when added together, ought to amount to about 99 or 99.5; and the small deficiency, always inevitable in such analyses, is put down as "loss," to make up the 100 parts.

SECTION V.

Quantitative Analysis of Copper Pyrites; consisting of Copper, Iron, Sulphur, and Silica (Quartz).

Determine the quantity of copper, iron, sulphur, and silica, in copper pyrites (Fe_2S_3,Cu_2S).

667. Boil 33.33 grains of the powdered mineral in strong nitric acid (see Fig. 75), until the sulphur which remains undissolved collects into yellowish globules. Dilute the acid mixture with two or three times its bulk of water; filter, and wash the insoluble residue (consisting of sulphur and silica) until the whole of the soluble matter is separated from it (627); reserve the insoluble residue for further examination (669).

(1.) *Estimation of the Sulphur.*

668. A portion of the sulphur will have become oxidized by the action of the nitric acid; and the sulphuric acid thus formed will be contained in the filtered liquid, in combination with the oxides of copper and iron.

Add chloride of barium to the solution as long as it causes a precipitate (647); boil the mixture; filter, wash well with hot water, and ignite the precipitate. From the weight of the sulphate of baryta thus obtained, that of the sulphur from which the sulphuric acid was derived, may be ascertained by the following calculation:—

$$\underbrace{\text{Atc. wt. of sulphate of baryta.}}_{116.5} : \underbrace{\text{Atc. wt. of sulphur.}}_{16} :: \underbrace{\text{Wt. of sulphate of baryta obtained.}}_{a} : \underbrace{\text{Wt. of sulphur dissolved.}}_{x}$$

699. The weight of the sulphur which resisted the action of the nitric acid (667) must now be estimated. The undissolved residue is to be thoroughly dried at 212°, and weighed: it is then gradually heated to redness in a counterpoised or weighed porcelain crucible, until the whole of the sulphur is burnt off, when it must be again weighed, the loss during ignition being of course the sulphur. This weight must be added to that already deduced from the sulphate of baryta (668), which, together, will give the quantity of sulphur contained in 33.33 grains of the mineral, or one-third of the per-centage.

(2.) *Estimation of the Silica.*

670. The siliceous matter is left after the expulsion of the sulphur, by ignition, from the residue insoluble in aqua regia.[1] The per-centage is obtained by multiplying by three.

(3.) *Estimation of the Copper.*

671. The solution filtered from the sulphate of baryta, (668), containing a slight excess of chloride of barium, is now treated with a slight excess of sulphuric acid, to remove the superfluous baryta, which is separated by filtration. The clear solution is diluted to about eight ounces with water, and thoroughly saturated with hydrosulphuric acid gas (655). The precipitated sulphide of copper is collected upon a filter, and rapidly washed with water containing hydrosulphuric acid, until a drop of the washings is no longer tinged black by ammonia; the filtrate and washings are reserved for further treatment

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[1] A trace of tin is occasionally found in the pyrites, and will be contained in this residue as peroxide (SnO_2). It may be detected by the blowpipe (118).

according to (672). The funnel containing the precipitated sulphide of copper is placed over a flask, the apex of the filter carefully perforated with a conical glass rod. and as much of the precipitate as possible washed through into the flask by means of a fine stream of water from a washing-bottle. The filter is then dried (631), incinerated, and the ashes added to the contents of the flask; add two drachms of strong nitric acid, and heat, at first gently, but finally to boiling (see Fig. 75), until the sulphur separates in yellow globules. Dilute the solution with two ounces of water, filter, or pour it clear off into a beaker, and determine the copper as oxide, according to the directions given at (645). From the weight of the oxide (CuO), that of the copper is calculated thus:—

$$\underbrace{\text{Atc. wt. of oxide of copper.}}_{39.8} : \underbrace{\text{Atc. wt. of copper.}}_{31.8} :: \underbrace{\text{Wt. of oxide obtained.}}_{a} : \underbrace{\text{Wt. of copper in 33.33 grs. of pyrites.}}_{x}$$

which, when multiplied by three, represents the percentage of copper in the mineral.

(4.) *Estimation of the Iron.*

672. The solution filtered from the sulphide of copper, must now be boiled to expel the excess of hydrosulphuric acid, and heated with a little nitric acid, for the purpose of peroxidizing the iron (241); ammonia is then added in slight excess; this throws down the iron as hydrated peroxide, which is to be filtered, washed, dried, ignited, and weighed. The weight of the iron contained in the precipitate is thus calculated:—

$$\underbrace{\text{Atc. wt. of peroxide of iron.}}_{80} : \underbrace{2\times\text{Atc. wt. of iron.}}_{56} :: \underbrace{\text{Wt. of oxide obtained.}}_{a} : \underbrace{\text{Wt. of iron contained in 33.33 grs. of pyrites.}}_{x}$$

673. The quantities thus obtained, should, when added together, amount to a fraction less than 100, the deficiency being, as before, set down as "loss":—

Copper	
Iron	
Sulphur	
Silica	
Loss	
	100.00

SECTION VI.

Quantitative Analysis of Newton's Fusible Alloy; consisting of Tin, Lead, and Bismuth.

Determine the quantity of tin, lead, and bismuth in Newton's fusible alloy.

(1.) *Estimation of the Tin.*

674. Twenty-five grains of the alloy are boiled with nitric acid diluted with an equal volume of water, until completely oxidized; the mixture is diluted with three or four times its bulk of water, and again heated to dissolve the nitrates of lead and bismuth; the residue of binoxide of tin is collected upon a filter, washed with hot water till the washings are no longer tinged by hydrosulphuric acid, dried, ignited (taking care to detach as much of the precipitate as possible from the filter, in order to avoid reduction), and weighed. The quantity of tin present is then calculated by the proportion:—

$$\overset{\text{Atc. wt. of binoxide of tin.}}{\underbrace{\qquad\qquad}_{75}} : \overset{\text{Atc. wt. of tin.}}{\underbrace{\qquad\qquad}_{59}} :: \overset{\text{Wt. of binoxide obtained.}}{\underbrace{\qquad\qquad}_{a}} : \overset{\text{Wt. of tin in 25 grs. of alloy.}}{\underbrace{\qquad\qquad}_{x}}$$

x, being multiplied by four, gives the per-centage of tin.

(2.) *Estimation of the Lead.*

675. The filtrate and washings from the binoxide of tin are evaporated to dryness, redissolved in about an ounce of water, a few drops of nitric acid being added to redissolve the basic nitrate of bismuth, and the solution is then mixed with an excess of dilute sulphuric acid over and above that required for complete precipitation; the mixture is allowed to stand for an hour, for the complete separation of the sulphate of lead, which is collected upon a small filter, washed[1] till the washings

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[1] Care must be taken to wash the edge of the filter thoroughly, or the sulphuric acid will cause it to char and fall to pieces when dry; it is advisable, after the washing with water is completed, to wash twice or thrice with alcohol (in which the precipitate is quite insoluble), in order to remove the sulphuric acid.

leave only a slight residue when evaporated upon a slip of glass, dried, carefully detached from the filter (which is incinerated separately), and weighed. The following proportion will give the amount of lead:—

$$\underbrace{\text{Atc. wt. of sulphate of lead.}}_{151.5} : \underbrace{\text{Atc. wt. of lead.}}_{103.5} :: \underbrace{\text{Wt. of sulphate of lead obtained.}}_{a} : \underbrace{\text{Lead in 25 grs. of alloy.}}_{x}$$

(3.) *Estimation of the Bismuth.*

676. The acid filtrate from the sulphate of lead is saturated with sulphuretted hydrogen, and the precipitated sulphide of bismuth, having been washed with water containing sulphuretted hydrogen, is rinsed into a flask (671), and boiled with dilute nitric acid; the separated sulphur, containing a little sulphide of bismuth, is filtered off, washed, and burnt in a small porcelain crucible at the lowest possible temperature; the residue left after the expulsion of the sulphur is dissolved in dilute nitric acid, and this solution having been mixed with the former, the whole is carefully evaporated to dryness in a porcelain dish, the residue dissolved in water, with a few drops of nitric acid, if necessary, and the solution mixed in a flask with an excess of sesquicarbonate of ammonia; the solution is digested for some time at a gentle heat, the precipitate collected upon a filter, washed, dried, ignited, and weighed, every care being taken to detach all the precipitate from the filter, which should be incinerated separately. The ignition converts the carbonate into the teroxide of bismuth, and the amount of the metal is therefore calculated by the following proportion:—

$$\underbrace{\text{Atc. wt. of teroxide of bismuth.}}_{234} : \underbrace{\text{Atc. wt. of bismuth.}}_{210} :: \underbrace{\text{Wt. of the teroxide obtained.}}_{a} : \underbrace{\text{Wt. of bismuth in 25 grs. of alloy.}}_{x}$$

The quantities per cent. of

Tin . . . .
Lead . . . .
and Bismuth . . . . . .

so obtained, should amount to nearly 100.

PART V.

REAGENTS.

CHAPTER I.

EXAMINATION AND USES OF REAGENTS.

677. The following is a list of the reagents, &c., usually employed in testing and analysis:—

Sulphuric acid, strong and dilute.
Hydrochloric acid.
Nitric acid.
*Nitrohydrochloric acid (aqua regia).
*Oxalic acid.
Acetic acid.
Tartaric acid.
Hydrosulphuric acid (sulphuretted hydrogen).
*Hydrofluosilicic acid.
Ammonia.
Hydrosulphate of ammonia.
Carbonate of ammonia.
Chloride of ammonium.
Oxalate of ammonia.
Phosphate of soda and ammonia (microcosmic salt).
Potash.
*Carbonate of potash.
Nitrate of potash.
*Iodide of potassium.
*Chromate of potash.
*Acetate of potash.
Cyanide of potassium.
Ferrocyanide of potassium (yellow prussiate of potash).
Ferridcyanide of potassium (red prussiate of potash).
Antimoniate of potash.
Carbonate of soda.
Phosphate of soda.
Sulphate of soda.
Borax.
Lime-water.
Sulphate of lime.
Chloride of calcium.
Chloride of barium.
Hydrate of baryta.
*Nitrate of baryta.
Sulphate of magnesia.
*Sulphate of manganese.
Perchloride of iron.
Sulphate of iron.
Granulated zinc.
Nitrate of cobalt.
Sulphate of copper.
*Ammonio-sulphate of copper
Acetate of lead.
*Subacetate of lead.
Nitrate of silver.
*Ammonio-nitrate of silver.
Perchloride of mercury.
Protochloride of tin.
*Perchloride of gold.
Bichloride of platinum.
*Sulphate of indigo.
Solution of starch.
*Black flux.
Distilled water.
Alcohol.
Litmus and turmeric paper.

* Those marked with an asterisk are not absolutely indispensable at the commencement.

678. Most of these substances, as they are met with in commerce, being always more or less impure; and as those even which are sold in the shops as pure reagents, are not unfrequently found, on examination, to be otherwise; it is always necessary, before taking a reagent into use, to ascertain by experiment whether it is of sufficient purity for the purposes for which it is intended. It may be stated as a general rule, that, when a chemical substance is required for use in analysis, it ought to be as nearly pure as possible; while, for many of the other operations of chemistry, the substances which are usually met with in commerce are sufficiently pure. The following brief remarks relative to the more common impurities of reagents, together with their principal uses, will probably be found useful to the student.

Sulphuric Acid (HO,SO_3).[1]

679. Sulphuric acid, as found in commerce, is never pure. The most common impurities are sulphate of lead (PbO,SO_3), nitric acid (NO_5), or binoxide of nitrogen (NO_2), and occasionally arsenic, and saline matters.

(*a*) If it contains the first, it will become turbid when diluted with four or five times its bulk of water, owing to the sulphate of lead, which is soluble in the strong acid, being insoluble in the dilute.

(*b*) Nitric acid, or the binoxide of nitrogen, is detected by boiling a small portion tinged with a solution of sulphate of indigo, when, if nitric acid is present, the blue color will disappear.[2]

(*c*) Arsenic may be detected by Marsh's test (282).

(*d*) Any fixed saline impurity remains as a residue when a few drops of the acid are evaporated on platinum foil.

680. The uses of sulphuric acid are very numerous. Besides being employed extensively in many branches of manufacture, it is used in the laboratory as a powerful decomposing agent; owing to its strong affinity for bases,

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[1] The strength of the acid is indicated by its specific gravity, which should be about 1.84. (See Appendix, Table I.)

[2] A more delicate test consists in pouring the acid slowly down to the bottom of a test-tube containing solution of sulphate of iron (361).

nearly all saline compounds are decomposed by it, and its solvent powers are also very great. It is often employed for the purpose of decomposing organic matter; also in the preparation of hydrogen, hydrosulphuric acid, and other gases; as a test for certain metals, and for many other purposes.

681. When *dilute* sulphuric acid is required, it is prepared by mixing together in a beaker, one part of the strong acid with four parts of distilled water, always *adding the acid to the water*, which should be kept constantly stirred, and allowing the precipitated sulphate of lead (if any) to subside, after which the clear liquid may be poured off.

Hydrochloric Acid (HCl).

682. This acid, in the form met with in commerce, is never pure, usually containing sulphurous acid, sulphuric acid, and chloride of iron, and occasionally free chlorine and chloride of arsenic.

(*a*) Evaporate a drop or two on a piece of glass; if pure, no residue is left.

(*b*) Dilute a portion with four or five times its bulk of distilled water, and add a drop of chloride of barium; if sulphuric acid is present, a white precipitate is produced.

(*c*) Add ammonia in excess, and hydrosulphate of ammonia; a black or green precipitate indicates iron.

(*d*) Add a little of the acid to water tinged with sulphate of indigo; if it contains free chlorine, the blue color is bleached.

(*e*) Arsenic may be detected by Marsh's test (282).

(*f*) Sulphurous acid may be detected by dissolving a fragment of zinc in the acid, when hydrosulphuric acid will be evolved, and may be recognized by paper moistened with acetate of lead (366).

683. The uses of hydrochloric acid are very numerous, especially in analysis, in which it is of constant value as a solvent for substances which are insoluble in water; many of the metals dissolve readily in it, forming soluble chlorides, and it is occasionally used to precipitate silver and mercury from their solutions.

When *dilute* hydrochloric acid is required, the strong acid may be diluted with about twice its bulk of water. The strong acid should have the specific gravity 1.20 (see Table III. in Appendix).

Nitric Acid (HO,NO_5).

684. Nitric acid, as met with in commerce, usually contains sulphuric and hydrochloric acids, and occasionally a little fixed saline matter.

(*a*) The latter may be detected by evaporating a few drops on a slip of glass, when any fixed impurities will be left.

Dilute a little of the acid with water, and divide it into two portions.

(*b*) To the first, add chloride of barium: if a white precipitate is produced, sulphuric acid is present (80).

(*e*) To the other, add nitrate of silver; a white precipitate, soluble in ammonia, indicates hydrochloric acid.

685. Nitric acid is used chiefly as a solvent for substances which are insoluble in water, especially some of the metals, which it readily oxidizes, and converts into nitrates, nearly all of which are soluble in water. It is also frequently employed to raise compounds to a higher state of oxidation, as in converting the protoxide of iron (FeO) into the peroxide (Fe_2O_3).

When *dilute* nitric acid is required, it may be prepared by mixing one part of the strong acid with two parts of distilled water. The specific gravity of the strong acid should be 1.45 (see Table II. in Appendix).

Nitrohydrochloric Acid (*Aqua Regia*).

686. This is always prepared when required, by mixing together strong nitric and hydrochloric acids, usually in the proportion of one part of nitric to four of hydrochloric. Its chief uses depend on its intense oxidizing or chlorinizing properties, whereby the most refractory metals, some of which resist the action of all other acids, are brought into solution.

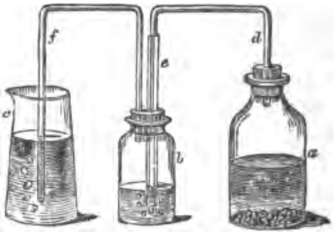
Hydrosulphuric Acid (HS). (*Sulphuretted Hydrogen.*)

687. This reagent, whether required in the gaseous form or in solution, is always prepared in the laboratory. Fragments of sulphide (sulphuret) of iron (FeS) are placed in a bottle, *a* (Fig. 98), together with three or four ounces of water; a little strong sulphuric acid is then added, which disengages the gas.

$$FeS + HO{,}SO_3 = FeO{,}SO_3 + HS.$$

The gas thus formed is passed through water contained in the second bottle, *b*, for the purpose of purifying it from any sulphuric acid and iron that may have been carried over mechanically, and is then conducted, by the bent tube *f*, into a bottle of distilled water, when an aqueous solution of the gas is required, or into a jar containing any solution which it is intended to act upon.

Fig. 98.



Sulphuretted-hydrogen Apparatus.

It must be borne in mind when experimenting with this gas, that it is not only highly offensive, but poisonous, and induces headache and nausea even when largely diluted with air: on this account it should be prepared either near a ventilating flue, or in the open air; never in a close room.

688. In most cases of mere testing, the aqueous solution is the most convenient form in which to apply it. The water should be saturated with the gas, of which it is capable of retaining in solution about two or three times its volume; this may be judged of by its strong disagreeable smell, resembling that of rotten eggs, and

by its giving a copious white precipitate of sulphur when treated with perchloride of iron (245). It should also be tested for iron, which it sometimes contains when carelessly prepared: if such is the case, it becomes dark-colored on the addition of ammonia, owing to the formation of sulphide of iron (238). The solution should not be kept long, as it is liable to decompose, unless carefully closed from the air, the oxygen of which combines with the hydrogen to form water (HO), while sulphur is deposited.[1]

689. When it is required to precipitate, by hydrosulphuric acid, *the whole* of any metal in a solution, it is necessary to pass the gas at once into it; and this should be continued until the liquid is saturated, which is known by removing the gas-delivering tube, and blowing away the superincumbent air; when, if it smells distinctly of the gas, the solution may be considered saturated, and the whole of the metal must have been converted into sulphide.

690. The important uses to which hydrosulphuric acid is applied, render it of great value in many processes of analysis. It precipitates many of the metals from their solutions as insoluble sulphides; and is one of the reagents employed in determining the class to which an unknown metal in solution belongs. It is also extensively used in quantitative analysis, on account of the perfect manner in which it separates the whole of many of the metals from their solutions. Hydrosulphuric acid is also sometimes useful as a deoxidizing agent, reducing metallic oxides in solution to a lower degree of oxidation, as the peroxide of iron to the protoxide; this property is owing to the facility with which it is decomposed, yielding up its hydrogen to the oxygen of the oxide, while the sulphur is usually set free (245).

Hydrofluosilicic Acid (HF,SiF_2).

691. In order to prepare this reagent, a mixture of equal parts of finely powdered fluor spar and sand is

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[1] In order to keep it as long as possible, the stopper should be greased, and the bottle kept inverted. When it is decomposed, the same (and not fresh) water should be again saturated with the gas, since all the free oxygen dissolved in it has been consumed.

heated with six parts of concentrated sulphuric acid in a large dry flask. The gaseous fluoride of silicon thus evolved, is conducted through a dry tube bent twice at right angles, into a glass filled with water, but containing at the bottom a sufficient quantity of mercury to cover the orifice of the delivery tube, in order to prevent its being choked by the gelatinous silica which is deposited as soon as the gas meets the water (401). The solution is filtered from the silica and is ready for use. It should give a decided crystalline precipitate with chloride of barium, but none with the chlorides of calcium and strontium.

692. Hydrofluosilicic acid is chiefly employed as a test for baryta.

Oxalic Acid (HO,C_2O_3).

693. Oxalic acid occasionally contains traces of nitric acid (which causes it to deliquesce in damp air, and to have a slight acid smell), and also fixed saline matter.

(*a*) The former may be detected by sulphate of iron (361).

(*b*) The latter is left as a fixed residue after ignition on platinum foil.

It may be easily purified by recrystallization.

The chief use to which oxalic acid is applied in analysis, is to precipitate lime from its solutions. (See also *Oxalate of ammonia.*) For use as a test, one part of the crystallized acid may be dissolved in ten parts of water.

Acetic Acid ($HO,C_4H_3O_3$).

694. This acid is often contaminated with one or more of the following substances: sulphuric, sulphurous, and hydrochloric acids, lead, and certain saline matters.

(*a*) Any fixed impurity may be detected by heating a little on a slip of glass.

(*b*) Add to a portion of the diluted acid, a solution of chloride of barium: if sulphuric acid is present, a white precipitate is thrown down.

(*c*) Test for sulphurous acid by (366).

(*d*) Nitrate of silver, added to the diluted acid, gives a white curdy precipitate, which is insoluble in nitric acid, if any hydrochloric acid is present.

(*e*) Neutralize a small portion with ammonia, and add hydrosulphuric acid or hydrosulphate of ammonia: if lead or any other metallic matter is present (except the alkalies and alkaline earths), a precipitate is produced.

Acetic acid is chiefly employed in the laboratory as a solvent, and for the purpose of acidifying solutions, in cases where hydrochloric and nitric acid would act prejudicially.

Tartaric Acid ($2HO,C_8H_4O_{10}$).

695. Tartaric acid sometimes contains a trace of lime and sulphuric acid, but is usually sufficiently pure for analytical purposes. The lime may be detected by neutralizing a portion with ammonia, and adding oxalate of ammonia; and the sulphuric acid by chloride of barium.

696. Tartaric acid is used as a test for potash, with which it forms a sparingly soluble bitartrate. Its property of preventing the precipitation of iron and some other metals by the alkalies (439), is occasionally made available in analysis. It should be kept in a solid state, and a solution made when required, as when kept in solution it soon becomes mouldy; for this purpose the crystallized acid may be dissolved in about three times its weight of water.

Ammonia (NH_3).

697. The liquid ammonia of the shops is generally sufficiently pure for most purposes of analysis; it sometimes, however, contains traces of carbonate, sulphate, and muriate of ammonia, and occasionally chloride of calcium. The carbonate is detected by adding lime water; the sulphate by supersaturating with dilute hydrochloric acid, and testing with chloride of barium; the chloride of ammonium may be detected by supersaturating with nitric acid, and adding nitrate of silver; and the lime (chloride of calcium) with oxalate of ammonia.

Ammonia is used chiefly for the purpose of neutralizing acid solutions, and for precipitating metallic oxides from their solutions, most of which are decomposed by it.

Its specific gravity should be about 0.88 (see Table VI. in Appendix).

Hydrosulphate of Ammonia (NH_4S,HS).

698. Hydrosulphate of ammonia is prepared by passing a stream of hydrosulphuric acid gas (687) through a solution of ammonia until it is saturated. To ascertain whether the saturation is complete, a few drops may be tested with sulphate of magnesia; if the ammonia is saturated, this gives no precipitate; but if any free ammonia is left, it throws down the hydrate of magnesia. When first prepared, the solution is almost colorless, but it gradually becomes yellow, owing to partial decomposition, the oxygen of the air combining with the hydrogen, while sulphur is set free, and remains dissolved; when this decomposition has taken place, the addition of an acid causes not only the evolution of hydrosulphuric acid, but also precipitates the dissolved sulphur (373).

699. Hydrosulphate of ammonia is much used, both in qualitative and quantitative analysis, chiefly for the purpose of precipitating certain metals from their solutions, and for separating the metals of the third class from the alkalies and alkaline earths.

Carbonate of Ammonia ($2NH_4O,3CO_2$).

700. The common carbonate of ammonia is a sesquicarbonate, or a compound of the neutral carbonate (NH_4O,CO_2) and the bicarbonate ($NH_4O,2CO_2$). When the neutral carbonate is required, and it is the best suited for most purposes of analysis, it may be prepared in solution by dissolving one part, by weight, of the sesquicarbonate, in three or four parts of water, and adding one part of liquid ammonia (sp. gr. 0.96). It is frequently employed in analysis, to precipitate some of the metals as carbonates: it is also used to neutralize acid solutions, and for other purposes.[1]

701. It is occasionally contaminated with traces of

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[1] A saturated solution of the sesquicarbonate, prepared by agitating an excess of the salt with cold water, is required for some purposes, as for the separation of tin and antimony.

animal oil, chloride of ammonium, sulphate of ammonia, and oxide of iron.

(*a*) Heat a small fragment on platinum foil: if any fixed saline impurity is present, it will be left after ignition; and if any charring takes place, it indicates the presence of animal matter.

(*b*) Supersaturate a little of the solution with nitric acid, and add to one portion a few drops of chloride of barium: a white precipitate, insoluble in nitric acid, indicates sulphuric acid.

(*c*) To the other portion of the acid solution, add nitrate of silver; if any chloride of ammonium is present, it will cause a white curdy precipitate.

(*d*) Add a little hydrosulphate of ammonia to the solution; a dark tinge indicates the presence of a trace of iron.

Chloride of Ammonium (NH_4Cl).

702. The sal ammoniac of commerce usually contains a considerable quantity of iron; and must therefore be purified, for analytical purposes, by crystallization. When pure, it should be entirely volatilized when heated on platinum foil, and its solution should not give any dark tinge with hydrosulphate of ammonia. One part of the salt should be dissolved in eight parts of water.

Oxalate of Ammonia (NH_4O,C_2O_3).

703. This salt, as met with in the shops, is sufficiently pure for all purposes of analysis. Like oxalic acid, it is employed chiefly for the purpose of precipitating lime from its solutions; for this purpose, it may be dissolved in about six times its weight of water.

Phosphate of Soda and Ammonia (*Microcosmic Salt*).
(NaO,NH_4O,HO,PO_5).

704. This salt occasionally contains traces of chloride of sodium, which may readily be detected by adding a few drops of nitrate of silver to a solution of the salt, acidified with nitric acid, when a curdy white precipitate indicates the presence of the chloride.

Microcosmic salt is used almost exclusively in blowpipe experiments; when heated, it is decomposed, the ammonia and water are expelled, and metaphosphate of soda (NaO,PO_5) is left.

Potash (*KO,HO*).

705. On account of its strong affinity for many substances, and its property of readily decomposing others, caustic potash is rarely found free from impurities. Those most commonly met with are organic matter, sulphate and carbonate of potash, chloride of potassium, silicic acid, alumina, and oxide of lead.[1]

(*a*) If organic matter is present, the solution of potash is more or less brown, and, on evaporation, leaves a brown residue.

(*b*) Sulphuric acid is detected by diluting the potash with water, supersaturating with nitric or hydrochloric acid, and adding chloride of barium, when, if it is present, the white insoluble sulphate is thrown down.

(*c*) If carbonic acid is present, it causes effervescence when the solution is supersaturated with hydrochloric acid.

(*d*) A little of the diluted solution is supersaturated with nitric acid, and tested with nitrate of silver; a white curdy precipitate, soluble in ammonia, indicates chloride of potassium.

(*e*) Neutralize a small portion with hydrochloric acid, and evaporate to dryness: if the residue is not wholly soluble in hydrochloric acid, silica is probably present.

(*f*) If alumina is present, it will be precipitated when the hydrochloric solution from (*e*) is treated with a *slight* excess of ammonia.

(*g*) If lead be present, the potash will give a dark precipitate or coloration with hydrosulphate of ammonia.

Potash is used chiefly for the purpose of precipitating some of the metallic oxides from their saline solutions, which it does on account of its strong affinity for the acids with which they were in combination.

$$CuO,SO_3 + KO,HO = KO,SO_3 + \mathrm{CuO,HO}.$$

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[1] When potash is kept in flint-glass bottles, it dissolves oxide of lead from the glass. German or green glass bottles are preferable.

It is employed also for neutralizing acid solutions, decomposing organic compounds, and many other purposes. A solution of potash having a specific gravity of about 1060, is of convenient strength for most analytical purposes. (See Table IV. in Appendix).

Carbonate of Potash (KO,CO_2).

706. This salt generally contains traces of sulphate and chloride, and occasionally silica and alumina.

(*a*) A solution, supersaturated with hydrochloric acid, and tested with chloride of barium, gives a white precipitate if any sulphuric acid is present.

(*b*) A solution acidified with nitric acid gives, with nitrate of silver, a white curdy precipitate, if it contains chloride of potassium.

(*c*) Neutralize a portion of the solution with hydrochloric acid, and evaporate to dryness; if the residue does not wholly dissolve when treated with hydrochloric acid, silica is probably present.

(*d*) If ammonia causes, in the hydrochloric solution, a white gelatinous precipitate, alumina is probably present.

Carbonate of potash is frequently employed to precipitate metallic oxides and carbonates from their soluble combinations, and for the purpose of neutralizing acid solutions.

Nitrate of Potash (KO,NO_5).

707. Nitrate of potash often contains traces of sulphate and chloride, and occasionally nitrates of soda and lime.

(*a*) and (*b*) The sulphate and chloride may be detected with chloride of barium and nitrate of silver.

(*c*) If lime is present, it causes a precipitate when the solution is treated with oxalate of ammonia.

(*d*) The presence of nitrate of soda causes the salt to deliquesce in a moist atmosphere.

708. It is used almost exclusively in the dry state, for the purpose of oxidizing substances which resist other methods of oxidation; this property is owing to the oxygen of the nitric acid being loosely combined, and at a high temperature readily yielded up to any substance

which has a strong affinity for it, such as sulphides, organic matters, &c.

Iodide of Potassium (KI).

709. Iodide of potassium is often adulterated with carbonate of potash; sulphate of potash and chloride of potassium are also often present. It should always be in the form of well defined (cubical) crystals, as the adulterated varieties are readily distinguishable by their imperfect crystalline form.

(*a*) Add a little dilute hydrochloric acid: if effervescence takes place, some carbonate is present.

(*b*) If sulphates are present, they may be detected by adding chloride of barium, which will, in that case, cause a white precipitate, insoluble in hydrochloric acid.

(*c*) Add a little nitrate of silver; this will cause a pale yellow precipitate of iodide of silver, together with chloride of silver, in case any soluble chloride is present. To separate them, filter the mixture, and after washing the precipitate, treat it with a slight excess of ammonia, which dissolves the chloride, and leaves the iodide undissolved; on neutralizing the ammoniacal solution with nitric acid, the appearance of a white curdy precipitate indicates the presence of a chloride.

Iodide of potassium is employed chiefly as a test for lead, mercury, and occasionally some of the other metals. For use as a reagent, one part of the salt may be dissolved in twenty parts of water.

Chromate of Potash (KO,CrO_3).

710. This salt occasionally contains traces of sulphate of potash, which is readily detected by precipitating a little of the solution with chloride of barium, and adding an excess of hydrochloric acid, which redissolves the chromate of baryta, while any sulphate remains insoluble.

It is employed as a test for several of the metallic oxides, with many of which it forms insoluble salts (chromates) of characteristic colors, as the chromate of lead, which is bright yellow. For use as a reagent it may be dissolved in ten times its weight of water.[1]

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[1] The bichromate ($KO,2CrO_3$) is a better salt to keep in solution, as the chromate attacks the glass of the bottles and renders it opaque.

Acetate of Potash ($KO,C_4H_3O_3$).

711. The commercial salt is liable to contain carbonate of potash, which causes it to effervesce when mixed with hydrochloric acid. Solution of acetate of potash should not be tinged by hydrosulphate of ammonia. The salt may be dissolved for use in three times its weight of water.

Cyanide of Potassium (KCy).

712. Cyanide of potassium is sometimes used in blowpipe experiments, and also as a liquid test. It should be white, and entirely soluble in water.

Ferrocyanide of Potassium ($K_2,FeCy_3$). (*Yellow Prussiate of Potash.*)

713. This salt, as met with in commerce, is sufficiently pure for the purposes of testing. It is employed as a test for the persalts of iron, with which it forms a deep blue precipitate of sesquiferrocyanide of iron, or Prussian blue. It gives characteristic precipitates, also, with some other metals. For use as a reagent, one part of the salt may be dissolved in fifteen or twenty parts of water.

Ferridcyanide of Potassium (K_3,Fe_2Cy_6). (*Red Prussiate of Potash.*)

714. It occasionally contains traces of the yellow prussiate, which is easily detected by the solution giving a blue precipitate with perchloride of iron (250). It is used as a test for the protosalts of iron, with which it forms a blue precipitate of ferridcyanide of iron, which is similar in appearance to that formed by ferrocyanide of potassium with the persalts. It may be dissolved in ten or fifteen parts of water.

Antimoniate of Potash (KO,SbO_5).

715. This substance seldom or never contains any impurity that can interfere with its action as a test for soda, which is the only use to which it is applied in the laboratory. One part of the ordinary antimoniate of commerce should be boiled with twenty parts of water and filtered.

The solution must be kept in a well stoppered bottle, and carefully excluded from the air, as the carbonic acid is liable to decompose it, and cause a precipitation of antimonic acid. Its solution should give a decided crystalline precipitate, with even a dilute solution of carbonate of soda.

Carbonate of Soda (NaO,CO_2).

716. The best method of preparing pure carbonate of soda, is to ignite the crystallized bicarbonate ($NaO,HO,2CO_2$), when the second equivalent of carbonic acid and the water are expelled, and pure anhydrous carbonate is left. The salt of commerce frequently contains a little sulphate and chloride, which may be detected in the manner already detailed (706 *a* and *b*). The more impure varieties contain also traces of sulphide of sodium, and sulphite and hyposulphite of soda. If sulphide of sodium be present, hydrosulphuric acid (recognized by paper moistened with acetate of lead) will be evolved on adding hydrochloric acid in excess. If sulphite of soda be present, the acid solution will smell of sulphurous acid, and will evolve hydrosulphuric acid when a fragment of zinc is dropped into it. The presence of hyposulphite of soda would cause the acid solution to become turbid when heated, at the same time disengaging sulphurous acid.

717. It is employed for the same purposes as carbonate of potash (706), also as a flux for the blowpipe, and for fusing with insoluble silicates,[1] &c. For use as a liquid reagent, one part of the crystallized salt may be dissolved in three parts of water.

Phosphate of Soda ($2NaO,HO,PO_5$).

718. This salt sometimes contains a little sulphate and chloride. To detect these impurities, add to one portion, in solution, chloride of barium, and to the other nitrate

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[1] The *carbonate of potash and soda*, prepared by incinerating Rochelle salt (tartrate of potash and soda) is generally used for the decomposition of silicates, being much more fusible than the simple carbonates. A mixture of 53 parts of dry carbonate of soda with 69 parts of dry carbonate of potash will answer the purpose.

of silver, and supersaturate both with nitric acid; if the precipitate does not entirely dissolve in either case, a sulphate or chloride is present.

It is employed chiefly as a test for magnesia, with which it forms, in the presence of ammoniacal salts, the double phosphate of magnesia and ammonia. For the purpose of testing, it may be dissolved in ten parts of water.

Borax (*Biborate of Soda*), ($NaO,2BO_3$).

719. Borax occasionally contains traces of sulphate and chloride, which may be detected in the same way as in the phosphate of soda (718). It is employed almost exclusively as a flux in blowpipe experiments, for which purpose it is admirably adapted; the second equivalent of boracic acid which it contains, exerts a strong affinity for bases at a high temperature, and is capable of displacing several acids from their combinations; it also forms many double compounds and mixtures which are readily fusible.

Lime Water (*CaO in water*).

720. This reagent is prepared by digesting hydrate of lime (CaO,HO) in cold distilled water for an hour or two, stirring the mixture occasionally, and when the undissolved portion of the lime has subsided, pouring off the clear solution. As it is liable to spoil when exposed to the air, owing to the absorption of carbonic acid, it should be kept in a well stoppered bottle.

721. Lime water should be sufficiently strong to turn the yellow color of turmeric instantly and decidedly brown; and, when tested with carbonate of soda, should throw down a copious white precipitate of carbonate of lime. It is used as a test for carbonic acid and some of the organic acids.

Sulphate of Lime (CaO,SO_3).

722. Sulphate of lime being very sparingly soluble in water, is always used in the form of a saturated solution, which is prepared by digesting the sulphate in hot water, stirring it occasionally, and filtering off the clear solution

from the undissolved portion. It is used chiefly for distinguishing baryta from strontia. The solution ought to give an immediate precipitate of sulphate of baryta, when tested with chloride of barium.

Chloride of Calcium (CaCl).

723. This substance occasionally contains a little free lime or hydrochloric acid, and traces of iron. The first are detected by test paper, and the last, if present, causes hydrosulphate of ammonia to throw down in the solution a black precipitate, or to impart a greenish tint to the liquid. As a reagent, chloride of calcium is employed chiefly in testing for some of the organic acids. It is also of great use in the laboratory as a drying agent, having so strong an affinity for water, that a moist gas passed over it is rapidly and completely deprived of its water. For this purpose the chloride need not be absolutely pure; it should not be fused, but merely dried, as the unfused is more porous, and consequently offers a larger amount of surface to any gas passed over it.

Chloride of Barium (BaCl).

724. Cloride of barium sometimes contains traces of iron and lime. It should not be discolored by hydrosulphate of ammonia, and, after being treated with a slight excess of sulphuric acid, and filtered, the clear solution should leave no fixed residue when evaporated on platinum foil; because the whole of the baryta is separated by the sulphuric acid, and any other fixed matter must be some impurity.

It is used chiefly for the purpose of testing for acids, especially sulphuric, with which it forms the insoluble sulphate of baryta. For use, one part of the salt may be dissolved in ten parts of water.

Hydrate of Baryta (BaO,HO).

725. In solution, hydrate of baryta is employed in order to separate magnesia from the alkalies; it is prepared by boiling the crystallized hydrate with water. The solution must be preserved from contact with the air, for the carbonic acid of the latter will precipitate the baryta as carbonate.

The dry hydrate is used in the decomposition of insoluble silicates in order to test them for alkalies; it should therefore be free from these latter, which may be ascertained by dissolving in dilute hydrochloric acid, precipitating the baryta by ammonia and carbonate of ammonia, with the aid of heat, filtering, evaporating to dryness, and igniting, when no residue should be left.

Nitrate of Baryta (BaO,NO_5).

726. Nitrate of baryta is liable to the same impurities as chloride of barium (724), and they may be detected in the same way. It should also be free from any chloride, which may be known by adding nitrate of silver. Its uses are the same as those of chloride of barium, for which it is occasionally substituted in cases when the addition of the chloride would interfere with the subsequent stages of an analysis, as when we have to test for chlorides in the same solution. For use, it may be dissolved in ten parts of water.

Sulphate of Magnesia (MgO,SO_3).

727. This salt is employed as a test for phosphoric acid; a cold, saturated solution is convenient for this purpose.

Sulphate of Manganese (MnO,SO_3).

728. A solution of this salt is employed in testing for hypochlorites.

Perchloride of Iron (Fe_2Cl_3).

729. This salt is liable to contain a little free acid, and traces of the protochloride ($FeCl$). The free acid is detected by adding a very little ammonia, when the first precipitate will disappear on shaking, if free acid be present. If any protosalt of iron is present, the solution gives a blue color with ferridcyanide of potassium (244). It is used as a test for some of the organic acids, and is also sometimes useful in the determination of phosphoric acid. It may be dissolved in five parts of water.[1]

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[1] It is usually purchased in the form of solution.

Sulphate of Iron (FeO,SO_3).

730. It is advisable to preserve this salt in the solid state, since its solution absorbs oxygen readily from the air; it is employed chiefly in testing for nitric and hydrocyanic acids.

Granulated Zinc.

731. In order to granulate it, scrap zinc is melted in an iron ladle, and poured from a height of eight or ten feet, in a slender stream, into a pail of water. It is liable to contain a little arsenic, which may be detected by Marsh's test (282).

The black scales which are left when commercial zinc is dissolved in dilute sulphuric or hydrochloric acid consist of lead; and a little tin may sometimes be detected in the hydrochloric solution by perchloride of mercury (345); these impurities, however, do not interfere with the common use of zinc for evolving hydrogen.

Nitrate of Cobalt (CoO,NO_5).

732. This reagent is used chiefly for the detection of alumina, zinc, magnesia, and some other substances, by means of the blowpipe. The solution employed for this purpose may contain one part of the salt dissolved in ten of water.

Sulphate of Copper (CuO,SO_3).

733. This salt is occasionally used as a test for arsenic and for other purposes: it may be dissolved in ten parts of water. The *ammonio-sulphate of copper* ($CuO,2NH_3,HO,SO_3$), which is also used in testing for arsenic, is prepared by adding ammonia to the solution of sulphate of copper, until the precipitate at first formed is nearly all redissolved, when the solution is filtered, and kept for use.

Acetate of Lead ($PbO,C_4H_3O_3$).

734. Acetate of lead is used as a test for several acids which form with oxide of lead insoluble salts. For testing, one part of the salt may be dissolved in ten parts of water.

Subacetate of Lead ($3PbO,C_4H_3O_3$).

735. The subacetate is prepared by boiling together equal weights of the neutral acetate and protoxide of lead (PbO) in water, and filtering the solution, which must be kept in a well-stoppered bottle, as it is easily decomposed when in contact with the air, owing to the strong affinity of the oxide of lead for carbonic acid. Both this and the neutral acetate are used in testing for hydrosulphuric acid, and for some of the other acids, especially carbonic.

Nitrate of Silver (AgO,NO_5).

736. This reagent is sometimes adulterated with nitrate of potash, and occasionally contains traces of copper and lead. When precipitated by a slight excess of hydrochloric acid, the filtered solution ought to leave no fixed residue when evaporated on platinum foil, as the whole of the silver would be thrown down, and any impurity would remain in solution. Copper is detected by adding ammonia in excess to the solution, when it will give the liquid a blue tinge. Nitrate of silver is used chiefly as a test for chlorine (chlorides and hydrochloric acid), and also for phosphoric and some of the other acids. For use as a reagent, one part of the salt may be dissolved in twenty parts of water.

737. The *ammonio-nitrate of silver* ($AgO,2NH_3,NO_5$), used as a test for arsenic is prepared by adding ammonia to a solution of the nitrate, until the precipitate at first thrown down is just redissolved.

Perchloride of Mercury ($HgCl$).

738. This is occasionally employed as a test for hydriodic and some other acids, and also for some kinds of organic matter: for this purpose it may be dissolved in twenty parts of water.

Protochloride of Tin ($SnCl$).

739. Protochloride of tin is prepared by boiling metallic tin in strong hydrochloric acid, care being taken that a portion of the metal remains undissolved, as other-

wise a little perchloride might be formed; the solution is then diluted with about four times its bulk of water. A few fragments of metallic tin should be kept in the solution, in order to prevent the formation of any perchloride.

740. Protochloride of tin is employed chiefly as a test for gold and mercury, and also as a deoxidizing agent, for which purpose it is well adapted, on account of its strong tendency to combine with oxygen or chlorine.

It occasionally contains traces of lead and iron, which may be detected by adding yellow hydrosulphate of ammonia in excess to the solution and applying heat, when, if pure, the precipitate is wholly redissolved, but if either of those metals is present, a black residue is left, since their sulphides are insoluble in the hydrosulphate.

Perchloride of Gold ($AuCl_3$).

741. This reagent is used almost exclusively as a test for the protosalts of tin, so that a very small quantity will be found sufficient for the purpose of testing. One part of the salt may be dissolved in thirty parts of water.[1]

Bichloride of Platinum ($PtCl_2$).

742. Bichloride of platinum is employed only as a test for potash and ammonia; it is prepared by dissolving scraps of platinum foil (previously cleaned by boiling in strong hydrochloric acid) in nitrohydrochloric acid, and evaporating this solution at a moderate heat to a syrupy consistence; it may be dissolved in so much water that the solution gives an immediate crystalline precipitate with a moderately dilute solution of chloride of potassium or chloride of ammonium.[2]

Sulphate of Indigo.

743. This substance may be prepared in solution, by dissolving a little indigo in strong sulphuric acid, and diluting the acid solution with water, so as to form a pale

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[1] Perchloride of gold and bichloride of platinum are usually purchased in solution.

[2] See note to (741).

blue liquid. It is used chiefly as a test for nitric acid and chlorine, by which it is decomposed, and its color discharged.

Solution of Starch ($C_{12}H_{10}O_{10}$).

744. This is made by gently boiling white starch with water. It is employed as a test for iodine, for which purpose small pieces of thread or paper may be steeped in the solution, dried, and kept for use.

Black Flux.

745. Black flux is an intimate mixture of carbonate of potash and finely divided charcoal, and is prepared by deflagrating in an iron spoon or crucible, a mixture of two parts of bitartrate of potash and one of nitre. It is used as a reducing flux in blowpipe experiments. For most purposes, an intimate mixture of three parts of dry carbonate of soda and one part of very finely powdered wood charcoal will answer equally well.

Distilled Water (HO).

746. Pure distilled water is prepared by carefully distilling any of the common kinds of water either in a still or retort, rejecting the first and last portions (62). For many purposes, rain water, when collected at a distance from towns or manufactories, and boiled and filtered, will be found sufficiently pure; but in analytical experiments, distilled water ought always to be used.

747. Before taking it into use, it should be tested with the following reagents:—

(*a*) Litmus and turmeric paper, for free acids and alkalies.

(*b*) Chloride of barium for sulphates.

(*c*) Nitrate of silver for chlorides. The mixture shortly becomes dark colored, especially if organic matter is present.

(*d*) Oxalate of ammonia for lime.

(*e*) Lime water for carbonic acid.

(*f*) Hydrosulphate of ammonia for any metals of the third or fourth class.

(*g*) When heated on a slip of glass it should leave the very slightest trace of solid residue.

Distilled water is used chiefly as a solvent, and for washing precipitates, besides many other purposes to which it is constantly applied.

Alcohol (C_4H_5O,HO).

748. The alcohol commonly used in chemical experiments should have a specific gravity of about 0.83, except in cases where absolute alcohol is required, when it should be 0.796.[1] When evaporated on glass, it should leave no residue, and should not change the color of litmus paper. It is used chiefly as a solvent, and for the purpose of facilitating the precipitation of substances which are less soluble in it than in water.

CHAPTER II.

METHODS OF IMPROVISING REAGENTS.

749. For the benefit of analysts who may be situated at a distance from towns where the necessary reagents can be purchased, a few suggestions are here offered as to the readiest methods of supplying the deficiency from sources which are generally available.

The *primary* chemicals, as they may be styled, with which it is indispensable that the analyst should be supplied are

Sulphuric Acid (Vitriol).
Carbonate of Soda (Washing Soda).
Chloride of Sodium (Common Salt).
Nitrate of Potash (Saltpetre).
Sulphur (Brimstone).
Ammonia (Hartshorn).
Lime.
Biborate of Soda (Borax).
Ferrocyanide of Potassium (Prussiate of Potash).
Carbonate of Sulphate of Baryta (Heavy Spar, Barytes).
Sulphate of Magnesia (Epsom Salts).
Litmus (Archil).

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[1] The strength of any sample may be inferred from its specific gravity by reference to table VII. in the Appendix.

With the aid of these, and of such common materials as can be everywhere procured, all the reagents absolutely necessary for qualitative analysis can be improvised.

Hydrochloric Acid (muriatic acid) would be prepared by distilling common salt with sulphuric acid (69).

Nitric Acid (aqua fortis), by distilling saltpetre with sulphuric acid (77).

Acetic Acid, by distilling common vinegar. Even vinegar itself might, on an emergency, be used instead of acetic acid, especially after boiling with a little wood charcoal, and filtering.

Tartaric Acid (not absolutely indispensable in analysis) might be made by boiling 4 parts of cream of tartar (argol, bitartrate of potash) with 1 part of chalk, adding to the mixture 2 parts of chalk dissolved in the least possible quantity of hydrochloric acid, filtering through a cloth, washing the precipitate (tartrate of lime) with water, and digesting at a gentle heat with $\frac{3}{5}$ths as much sulphuric acid as there was originally of cream of tartar, diluted with 6 parts of water. The insoluble sulphate of lime is strained off, and the solution crystallized by slow evaporation.

Hydrosulphuric Acid.—The sulphide of iron required for its preparation is made by throwing a mixture of 10 parts of iron filings with 7 parts of sulphur, by small portions at a time, into a red-hot crucible. The hydrosulphuric acid is then prepared according to (687).

If crude antimony ore (SbS_3) is procurable, hydrosulphuric acid may be prepared from it by heating with dilute hydrochloric acid in a flask, the gas being washed by passing through water.

A mixture of equal weights of sulphur and tallow, gently heated, will also evolve hydrosulphuric acid.

Ammonia.—The most portable form for this reagent is sal-ammoniac, from which it may be prepared by (72). Common smelling salts (Preston salts, carbonate of ammonia) might be substituted for the sal-ammoniac.

Carbonate of Ammonia.—Common smelling salts may be employed. Or if sal-ammoniac and carbonate of soda be distilled with a little water, a solution of carbonate of ammonia would be obtained. Putrefied urine is an excellent source from which to distil carbonate of ammonia.

Potash may be obtained by dissolving the carbonate of potash (common potashes, pearlash) in ten parts of water, and adding, by degrees, to the boiling solution, one part of quick-lime, slaked and mixed with water. Allow the mixture to settle in a covered vessel, and draw off the clear solution of potash. The carbonate of potash may be extracted from wood ashes (especially those of young shoots and leaves) by treatment with water; or from calcined argol (cream of tartar); or from the lees of wine, after calcination. Soda is a good substitute for potash, and may be prepared from carbonate of soda (common washing soda, soda-ash) by the process described above.

Lime (quick-lime) is easily procurable by calcining egg-shells, oyster-shells, chalk, or marble in an open fire.

Sulphate of lime is to be procured almost everywhere as *plaster of Paris.* It may easily be made by adding chalk to diluted sulphuric acid till it is no longer acid.

Chloride of barium may be prepared from the carbonate (witherite) or from the sulphate (heavy spar), both of which are used by color makers and others under the name of barytes. From the former the chloride of barium is made by dissolving in dilute hydrochloric acid and crystallizing. The sulphate of baryta is mixed with one-fourth its weight of charcoal and enough oil to form a paste; strongly calcined in a closed crucible, and the resulting sulphide of barium dissolved in hydrochloric acid, filtered, and crystallized.

Perchloride of iron (muriate of iron) is made by dissolving iron in hydrochloric acid mixed with one-fourth of nitric acid, and expelling the excess of acid by evaporation.

Sulphate of iron (copperas, green vitriol) is made by dissolving iron in dilute sulphuric acid and crystallizing.

Acetate of lead (sugar of lead) may be made by leaving scraps of lead (or shot) in an open bottle, partly covered with vinegar, until the latter is distinctly sweet, when water may be added, and the solution filtered.

Nitrate of silver (lunar caustic) is obtained by dissolving ordinary silver in nitric acid, evaporating to dryness in a dish, heating moderately for some time till the blue (nitrate of copper) is converted into black (oxide), dissolving in water, and filtering.

Hydrosulphate of ammonia may be made with hydrosulphuric acid and ammonia (698).

Chloride of ammonium (sal-ammoniac), by neutralizing hydrochloric acid with ammonia or carbonate of ammonia.

Oxalate of ammonia is prepared by neutralizing oxalic acid with ammonia or carbonate of ammonia. The acid is obtained by dissolving one part of sugar in eight parts of ordinary nitric acid and evaporating gently to crystallization.

Phosphate of soda is generally procurable at the druggists. A substitute for it is phosphate of ammonia, prepared by mixing three parts of bone-ash (bones burnt white in an open fire) with two parts of strong sulphuric acid and twenty of water, allowing the mixture to stand for a day or two, straining off the sulphate of lime, and mixing the solution with ammonia or carbonate of ammonia till it is alkaline. The solution of phosphate of ammonia is then filtered off, and concentrated by evaporation.

Nitrate of potash is of course readily improvised by neutralizing nitric acid with potash or carbonate of potash and crystallizing.

Cyanide of potassium is made by fusing eight parts of well dried ferrocyanide of potassium with three parts of dry carbonate of potash in a covered crucible till a drop on the end of an iron rod is white, or nearly so, on cooling.

Antimoniate of potash may be prepared by adding powdered antimony (or crude antimony) to nitre melted in a crucible as long as brisk deflagration ensues. The solution is then prepared from this mass as in (715.)

Chloride of calcium is prepared by neutralizing dilute hydrochloric acid with chalk.

Hydrate of baryta.—To prepare the hydrate of baryta, powdered chloride of barium may be added to a strong boiling solution of (caustic) potash or soda, and the solution filtered while hot; on cooling it will deposit crystals of hydrate of baryta, which may be purified by recrystallization. Or the carbonate of baryta (or sulphate of baryta after calcination with charcoal) may be dissolved in dilute nitric acid, the solution evaporated, and the residue of nitrate of baryta calcined in a crucible or iron pot; anhydrous baryta is left, which becomes hydrate in contact with water.

APPENDIX.

WEIGHTS AND MEASURES.

Apothecaries' Weight.

Pound.		Ounces.		Drachms.		Scruples.		Grains.		French Grammes.
1	=	12	=	96	=	288	=	5760	=	372.96
		1	=	8	=	24	=	480	=	31.08
				1	=	3	=	60	=	3.885
						1	=	20	=	1.295
								1	=	0.0647

Avoirdupois Weight.

Pound.		Ounces.		Drachms.		Grains.		French Grammes.
1	=	16	=	256	=	7000	=	453.25
		1	=	16	=	437.5	=	28.328
				1	=	27.343	=	1.77

Imperial Measure.

Gallon.		Pints.		Fluid Ounces.		Fluid Drachms.		Minims.
1	=	8	=	160	=	1280	=	76,800
		1	=	20	=	160	=	9,600
				1	=	8	=	480
						1	=	60

Weight of Water at 62°, contained in the Imperial Gallon, &c.

			Grains.
1	Imperial Gallon . . .	. = . .	70,000
1	“ Pint . . .	. = . .	8,750
1	“ Fluid Ounce .	. = . .	437.5
1	“ Fluid Drachm .	. = . .	54.7
1	“ Minim . . .	. = . .	0.91

Cubic Inches contained in the Imperial Gallon, &c.

				Cubic Inches.
1	Imperial	Gallon	=	277.276
1	"	Pint	=	34.659
1	"	Fluid Ounce	=	1.7329
1	"	Fluid Drachm	=	0.2166
1	"	Minim	=	0.0036

FRENCH WEIGHTS AND MEASURES.

Measures of Length.

		English Inches.		Mil.	Fur.	Yds.	Feet.	In.
Millimetre	=	.03937						
Centimetre	=	.39371						
Decimetre	=	3.93710						
Metre	=	39.37100						
Decametre	=	393.71000	=	0	0	10	2	9.7
Hecatometre	=	8937.10000	=	0	0	109	1	1
Kilometre	=	39371.00000	=	0	4	213	4	10.2
Myriometre	=	393710.00000	=	6	1	156	0	6

Measures of Capacity.

				English Imperial Measure.				
		Cubic Inches.		Gall.	Pints.	F.oz.	F.drms.	Min.
Millilitre	=	.06102	=	0	0	0	0	16.3
Centilitre	=	.61028	=	0	0	0	2	43
Decilitre	=	6.10280	=	0	0	3	3	2
Litre	=	61.02800	=	0	1	15	1	43
Decalitre	=	610.28000	=	2	1	12	1	16
Hecatolitre	=	6102.80000	=	22	0	1	4	48
Kilolitre	=	61028.00000	=	220	0	12	6	24
Myriolitre	=	610280.00000	=	2200	7	13	4	48

Measures of Weight.

				Avoirdupois.		
		English Grains.		lbs.	Oz.	Drms.
Milligramme	=	.0154				
Centigramme	=	.1543				
Decigramme	=	1.5432				
Gramme	=	15.4323				
Decagramme	=	154.3234	=	0	0	5.65
Hecatogramme	=	1543.2348	=	0	3	8.5
Kilogramme	=	15432.348	=	2	3	5
Myriogramme	=	154323.480	=	22	1	2

Showing the behavior of Solutions of the Metals with Hydrosulphuric Acid, Hydrosulphate of Ammonia, and Carbonate of Ammonia, employed successively (Dr. Will).—(The rarer metals are printed in italics.)

Elements precipitated from their acid solutions by Hydrosulphuric Acid, as Sulphides.				Bodies precipitated by Hydrosulphate of Ammonia.					Bodies not precipitated by Hydrosulphuric Acid, or Hydrosulphate of Ammonia. In the presence of Muriate of Ammonia, on addition of Carbonate of Ammonia, are	
Soluble in Hydrosulphate of Ammonia, and reprecipitated by Hydrochloric Acid.		Insoluble in Hydrosulphate of Ammonia.		As Sulphides.		As Oxides.		As Salts.	precipitated.	not precipitated.
Antimony.	Orange.	Mercury .	Blackish or Brownish black.	Nickel . .	Black.	Aluminum	Soluble in potash.	Baryta, Strontia, Lime.	Barium.	Magnesium.
Arsenic .	Yellow.	Silver . . .		Cobalt . .		*Glucinum*		in combination with phosphoric, boracic, oxalic, and some other acids.	Strontium.	Potassium.
Tin . . .		Lead . . .		Manganese	Flesh-colored.	Chromium			Calcium.	Sodium.
Gold . .	Black.	Bismuth .		Iron	Black.	*Thorinum*	Insoluble in potash.			*Lithium.*
Platinum		Copper . .		Zinc	White.	*Yttrium*		Magnesia.		Ammonium.
Iridium .		*Cadmium*.	Yellow.	*Uranium*	Brownish black.	*Cerium*		in combination with phosphoric acid.		
Molybdenum.	Brown.	*Palladium.*	Brownish black.			*Zirconium*				
		Rhodium .				*Titanium*				
		Osmium . .				*Tantalum*				

TABLE I.

Showing the Quantity of Oil of Vitriol (HO,SO_3), *of sp. gr.* 1.8485, *and of Anhydrous Acid* (SO_3) *in* 100 *Parts of dilute Sulphuric Acid, of different Specific Gravities* (*Ure*).

Liquid Acid.	Sp. Gr.	Dry Acid.	Liquid Acid.	Sp. Gr.	Dry Acid.
100	1.8485	81.54	59	1.4760	48.11
99	1.8475	80.72	58	1.4660	47.29
98	1.8460	79.90	57	1.4560	46.48
97	1.8439	79.09	56	1.4460	45.66
96	1.8410	78.28	55	1.4360	44.85
95	1.8376	77.46	54	1.4265	44.03
94	1.8336	76.65	53	1.4170	43.22
93	1.8290	75.83	52	1.4073	42.40
92	1.8233	75.02	51	1.3977	41.58
91	1.8179	74.20	50	1.3884	40.77
90	1.8115	73.39	49	1.3788	39.95
89	1.8043	72.57	48	1.3697	39.15
88	1.7962	71.75	47	1.3612	38.32
87	1.7870	70.94	46	1.3530	37.51
86	1.7774	70.12	45	1.3440	36.69
85	1.7673	69.31	44	1.3345	35.88
84	1.7570	68.49	43	1.3255	35.06
83	1.7465	67.68	42	1.3165	34.25
82	1.7360	66.86	41	1.3080	33.43
81	1.7245	66.05	40	1.2999	32.61
80	1.7120	65.23	39	1.2913	31.80
79	1.6993	64.42	38	1.2826	30.98
78	1.6870	63.60	37	1.2740	30.17
77	1.6750	62.78	36	1.2654	29.35
76	1.6630	61.97	35	1.2572	28.54
75	1.6520	61.15	34	1.2490	27.72
74	1.6415	60.34	33	1.2409	26.91
73	1.6321	59.82	32	1.2334	26.09
72	1.6204	58.71	31	1.2260	25.28
71	1.6090	57.89	30	1.2184	24.46
70	1.5975	57.08	29	1.2108	23.65
69	1.5868	56.26	28	1.2032	22.83
68	1.5760	55.45	27	1.1956	22.01
67	1.5648	54.63	26	1.1876	21.20
66	1.5503	53.82	25	1.1792	20.38
65	1.5390	53.00	24	1.1706	19.57
64	1.5280	52.18	23	1.1626	18.75
63	1.5170	51.37	22	1.1549	17.94
62	1.5066	50.55	21	1.1480	17.12
61	1.4960	49.74	20	1.1410	16.31
60	1.4860	48.92	19	1.1330	15.49

TABLE I.—*Continued.*

Liquid Acid.	Sp. Gr.	Dry Acid.	Liquid Acid.	Sp. Gr.	Dry Acid.
18	1.1246	14.68	9	1.0614	7.34
17	1.1165	13.84	8	1.0544	6.52
16	1.1090	13.05	7	1.0477	5.71
15	1·1019	12.23	6	1.0405	4.89
14	1.0953	11.41	5	1.0336	4.08
13	1.0887	10.60	4	1.0268	3.26
12	1.0809	9.78	3	1.0206	2.446
11	1.0743	8.97	2	1.0140	1.63
10	1.0682	8.15	1	1.0074	0.8154

TABLE II.

Showing the Quantity of Real or Anhydrous Nitric Acid (NO_5) *in* 100 *Parts of Liquid Acid, of different Specific Gravities* (*Ure*).

Specific Gravity.	Real Acid in 100 parts of the Liquid.	Specific Gravity.	Real Acid in 100 parts of the Liquid.	Specific Gravity.	Real Acid in 100 parts of the Liquid.
1.5000	79.700	1.4147	58.978	1.2826	38.256
1.4980	78.903	1.4107	58.181	1.2765	37.459
1.4960	78.106	1.4065	57.384	1.2705	36.662
1.4940	77.309	1.4023	56.587	1.2644	35.865
1.4910	76.512	1.3978	55.790	1.2583	35.068
1.4880	75.715	1.3945	54.993	1.2523	34.271
1.4850	74.918	1.3882	54.196	1.2462	33.474
1.4820	74.121	1.3833	53.399	1.2402	32.677
1.4790	73.324	1.3783	52.602	1.2341	31.880
1.4760	72.527	1.3732	51.805	1.2277	31.083
1.4730	71.730	1.3681	51.068	1.2212	30.286
1.4700	70.933	1.3630	50.211	1.2148	29.489
1.4670	70.136	1.3579	49.414	1.2084	28.692
1.4640	69.339	1.3529	48.617	1.2019	27.895
1.4600	68.542	1.3477	47.820	1.1958	27.098
1.4570	67.745	1.3427	47.023	1.1895	26.301
1.4530	66.948	1.3376	46.226	1.1833	25.504
1.4500	66.155	1.3323	45.429	1.1770	24.707
1.4460	65.354	1.3270	44.632	1.1709	23.910
1.4424	64.557	1.3216	43.835	1.1648	23.113
1.4385	63.760	1.3163	43.038	1.1587	22.316
1.4346	62.963	1.3110	42.241	1.1526	21.519
1.4306	62.166	1.3056	41.444	1.1465	20.722
1.4269	61.369	1.3001	40.647	1.1403	19.925
1.4228	60.572	1.2947	39.850	1.1345	19.128
1.4189	59.775	1.2887	39.053	1.1286	18.331

TABLE II.—*Continued.*

Specific Gravity.	Real Acid in 100 parts of the Liquid.	Specific Gravity.	Real Acid in 100 parts of the Liquid.	Specific Gravity.	Real Acid in 100 parts of the Liquid.
1.1227	17.534	1.0764	11.158	1.0375	5.579
1.1168	16.737	1.0708	10.361	1.0320	4.782
1.1109	15.940	1.0651	9.564	1.0267	3.985
1.1051	15.143	1.0595	8.767	1.0212	3.188
1.0993	14.346	1.0540	7.970	1.0159	2.391
1.0935	13.549	1.0485	7.173	1.0106	1.594
1.0878	12.752	1.0430	6.376	1.0053	0.797
1.0821	11.955				

TABLE III.

Showing the Quantity of Anhydrous Hydrochloric Acid (HCl) *in the Liquid Acid of different Specific Gravities* (*Ure*).

Acid of 1.20 in 100.	Specific Gravity.	Chlorine.	Hydrochloric Gas.	Acid of 1.20 in 100.	Specific Gravity.	Chlorine.	Hydrochloric Gas.
100	1.2000	39.675	40.777	73	1.1473	28.964	29.767
99	1.1982	39.278	40.369	72	1.1452	28.567	29.359
98	1.1964	38.882	39.961	71	1.1431	28.171	28.951
97	1.1946	38.485	39.554	70	1.1410	27.772	28.544
96	1.1928	38.089	39.146	69	1.1389	27.376	28.136
95	1.1910	37.692	38.738	68	1.1369	26.979	27.728
94	1.1893	37.296	38.330	67	1.1349	26.583	27.321
93	1.1875	36.900	37.923	66	2.1328	26.186	26.913
92	1.1857	36.503	37.516	65	1.1308	25.789	26.505
91	1.1846	36.107	37.108	64	1.1287	25.392	26.098
90	1·1822	35.707	36.700	63	1.1267	24.996	25.690
89	1.1802	35.310	36.292	62	1.1247	24.599	25.282
88	1.1782	34.913	35.884	61	1.1226	24,202	24.874
87	1.1762	34.517	35.476	60	1.1206	23.805	24.466
86	1.1741	34.121	35.068	59	1.1185	23.408	24.058
85	1.1721	33.724	34.660	58	1.1164	23.012	23.650
84	1.1701	33.328	34.252	57	1.1143	22.615	23.242
83	1.1681	32.931	33.845	56	1.1123	22.218	22.834
82	1.1661	32.535	33.437	55	1.1102	21.822	22.426
81	1.1641	32.136	33.029	54	1.1082	21.425	22.019
80	1.1620	31.743	32.621	53	1.1061	21.023	21.611
79	1.1599	31.343	32.213	52	1.1041	20.632	21.203
78	1.1578	30.946	31.805	51	1.1020	20.235	20.796
77	1.1557	30.550	31.398	50	1.1000	19.837	20.388
76	1.1536	30.153	30.990	49	1.0980	19.440	19.980
75	1.1515	29.757	30.582	48	1.0960	19.044	19.572
74	1.1494	29.361	30.174	47	1.0939	18.647	19.165

TABLE III.—*Continued.*

Acid of 1.20 in 100.	Specific Gravity.	Chlorine.	Hydrochloric Gas.	Acid of 1.20 in 100.	Specific Gravity.	Chlorine.	Hydrochloric Gas.
46	1.0919	18.250	18.757	23	1.0457	9.126	9.379
45	1.0899	17.854	18.349	22	1.0437	8.729	8.971
44	1.0879	17.427	17.941	21	1.0417	8.332	8.563
43	1.0859	17.060	17.534	20	1.0397	7.935	8.155
42	1.0838	16.664	17.126	19	1.0377	7.538	7.747
41	1.0818	16.267	16.718	18	1.0357	7.141	7.340
40	1.0798	15.870	16.310	17	1.0337	6.745	6.932
39	1.0778	15.474	15.902	16	1.0318	6,348	6.524
38	1.0758	15.077	15.494	15	1.0298	5.951	6.116
37	1.0738	14.680	15.087	14	1.0279	5.554	5.709
36	1.0718	14.284	14.679	13	1.0259	5.158	5.301
35	1.0697	13.887	14.271	12	1.0239	4.762	4.893
34	1.0677	13.490	13.863	11	1.0220	4.365	4.486
33	1.0657	13.094	13.456	10	1.0200	3.998	4.078
32	1.0637	12.597	13.049	9	1.0180	3.571	3.670
31	1.0617	12.300	12.641	8	1.0160	3.174	3.262
30	1.0597	11.903	12.233	7	1.0140	2.778	2.854
29	1.0577	11.506	11.825	6	1.0120	2.381	2.447
28	1.0557	11.109	11.418	5	1.0110	1.984	2.039
27	1.0537	10.712	11.010	4	1.0080	1.588	1.631
26	1.0517	10.316	10.602	3	1.0060	1.191	1.224
25	1.0497	9.919	10.194	2	1.0040	0.795	0.816
24	1.0477	9.522	9.786	1	1.0020	0.397	0.408

TABLE IV.

Showing the Quantity of Anhydrous Potash (KO) in Solutions of different Specific Gravities (Dalton).

Specific Gravity.	Potash per cent.	Boiling Point.	Specific Gravity.	Potash per cent.	Boiling Point.
1.63	51.2	329°	1.33	26.3	229°
1.60	46.7	290	1.28	23.4	224
1.52	42.9	276	1.23	19.5	220
1.47	39.6	265	1.19	16.2	218
1.44	36.8	255	1.15	13.0	215
1.42	34.4	246	1.11	9.5	214
1.39	32.4	240	1.06	4.7	213
1.36	29.4	234			

TABLE V.

Showing the Quantity of Anhydrous Soda (NaO) in Solutions of different Specific Gravities (Dalton).

Specific Gravity.	Soda per cent.	Boiling Point.	Specific Gravity.	Soda per cent.	Boiling Point.
2.00	77.8	—°	1.40	29.0	242°
1.85	63.6	600	1.36	26.0	235
1.72	53.8	400	1.32	22.0	228
1.63	46.6	300	1.29	19.0	224
1.56	41.2	280	1.23	16.0	220
1.50	36.8	265	1.18	13.0	217
1.47	34.0	255	1.12	9.8	214
1.44	31.0	248	1.06	4.7	213

TABLE VI.

Showing the Quantity of Ammoniacal Gas (NH_3) *in Aqueous Solutions of different Specific Gravities (Dalton).*

Specific Gravity.	Grains of Ammonia in 100 grains of the liquid.	Boiling Points.	Volumes of gas in one volume of the liquid.
.850	35.3	26°	494
.860	32.6	38	456
.870	29.9	50	419
.880	27.3	62	382
.890	24.7	74	346
.900	22.2	86	311
.910	19.8	98	277
.920	17.4	110	244
.930	15.1	122	211
.940	12.8	134	180
.950	10.5	146	147
.960	8.3	158	116
.970	6.2	173	87
.980	4.4	187	58
.990	2.0	196	28

TABLE VII.

Showing the Quantity of Absolute Alcohol (C_4H_5O,HO) *contained in Diluted Alcohol of different Specific Gravities* (*Fownes*).

Sp. Gr. at 60°.	Percentage of real alcohol.	Sp. Gr. at 60°.	Percentage of real alcohol.	Sp. Gr. at 60°.	Percentage of real alcohol.
0.9991	0.5	0.9511	34	0.8769	68
0.9981	1	0.9490	35	0.8745	69
0.9965	2	0.9470	36	0.8721	70
0.9947	3	0.9452	37	0.8696	71
0.9930	4	0.9434	38	0.8672	72
0.9914	5	0.9416	39	0.8649	73
0.9898	6	0.9396	40	0.8625	74
0.9884	7	0.9376	41	0.8603	75
0.9869	8	0.9356	42	0.8581	76
0.9855	9	0.9335	43	0.8557	77
0.9841	10	0.9314	44	0.8533	78
0.9828	11	0.9292	45	0.8508	79
0.9815	12	0.9270	46	0.8484	80
0.9802	13	0.9249	47	0.8459	81
0.9789	14	0.9228	48	0.8434	82
0.9778	15	0.9206	49	0.8408	83
0.9766	16	0.9184	50	0.8382	84
0.9753	17	0.9160	51	0.8357	85
0.9741	18	0.9135	52	0.8331	86
0.9728	19	0.9113	53	0.8305	87
0.9716	20	0.9090	54	0.8279	88
0.9704	21	0.9069	55	0.8254	89
0.9691	22	0.9047	56	0.8228	90
0.9678	23	0.9025	57	0.8199	91
0.9665	24	0.9001	58	0.8172	92
0.9652	25	0.8979	59	0.8145	93
0.9638	26	0.8956	60	0.8118	94
0.9623	27	0.8932	61	0.8089	95
0.9609	28	0.8908	62	0.8061	96
0.9593	29	0.8886	63	0.8031	97
0.9578	30	0.8863	64	0.8001	98
0.9560	31	0.8840	65	0.7969	99
0.9544	32	0.8816	66	0.7938	100
0.9528	33	0.8793	67		

TABLE VIII.

Showing the Specific Gravities of Mixtures of Ether and Alcohol in different proportions (Dalton).

Specific Gravity.	Ether.	Alcohol (sp. gr. 830).	Specific Gravity.	Ether.	Alcohol (sp. gr. 830).
724	100	0	792	40	60
732	90	10	804	30	70
744	80	20	816	20	80
756	70	30	828	10	90
768	60	40	830	0	100
780	50	50			

TABLE IX.

Showing the Solubility of Salts.[1]

Bases. Acids.	Sulphuric.	Phosphoric.	Boracic.	Carbonic.	Silicic.	Arsenious.	Arsenic.	Hydrochloric.	Hydriodic.	Hydrosulphuric.	Nitric.	Chloric.	Oxalic.	Tartaric.	Citric.	Malic.	Succinic.	Benzoic.	Acetic.	Formic.
Potash	1....	1	1	1	1 & 3	1	1....	1....	1	1....	1....	1....	1....	1 & 2	1....	1....	1....	1....	1....	1
Soda	1....	1	1	1	1..3	1	1....	1....	1	1....	1....	1....	1....	1....	1....	1....	1....	1....	1....	1
Ammonia	1....	1	1	1	....	1	1....	1....	1	1....	1....	1....	1....	1 & 2	1....	1....	1....	1....	1....	1
Magnesia	1....	2	2	2	2 & 3	2	2....	1....	1	....	1....	1....	2....	1..2	1....	1..2	1....	1....	1....	1
Lime	1.2.3	2	2	2	2 & 3	2	2....	1....	1	1...2	1....	1....	2....	2....	2....	1 & 2	2....	1..2	1....	1
Baryta	3....	2	2	2	2 & 3	2	2....	1....	1	1....	1....	1....	2....	2....	2....	1....	1..2	1....	1....	1
Strontia	3....	2	2	2	2 & 3	2	2....	1 ...	1	1....	1....	1....	2....	2....	1....	1....	1....	1....	1....	1
Alumina	1....	2	2	...	2 & 3	2	2....	1....	...	....	1....	1....	2....	1....	1 & 2	2....	....	1....	1....	1..2
Ox. Chromium	1....	2	2	2	2 & 3	...	1....	1....	...	....	1....	1....	1....	1....	1....	....	1....	....	1....	
Ox. Zinc	1....	2	2	2	2 & 3	...	1 & 2	1....	1	2....	1....	1....	2....	2....	2....	1..2	1....	1....	1....	1
Ox. Manganese	1....	2	2	2	2 & 3	...	2....	1....	1	2....	1....	1....	2....	1..2	1....	1....	1....	1....	1....	1
Protox. Iron	1....	2	2	2	2 & 3	2	2....	1....	1	2 ...	1....	1....	1..2	1..2	1....	1....	1....	....	1....	1
Perox. Iron	1....	2	2	...	2 & 3	...	2....	1....	1	2....	1....	1....	1..2	1....	1....	1....	2....	2....	1....	1
Ox. Nickel	1....	2	2	2	2 & 3	2	2....	1....	...	2....	1....	1....	2....	2....	2....	....	2....	1....	1....	1
Ox. Cobalt	1....	2	2	2	2 & 3	2	2....	1....	...	2....	1....	1....	2....	1....	....	....	2....	1....	1....	1..2
Ox. Antimony	1..2	2	...	...	....	2	2....	1 & 2	...	2....	....	....	1..2	1....	....	....	....	1....	1....	
Subox. Mercury	1..2	2	...	2	....	2	2....	2..3	2	3....	1 & 2	1 & 2	2....	1..2	2....	1....	2....	2....	1..2	1
Perox. Mercury	1....	2	...	2	....	2	2....	1.. .	2	3....	1....	1....	2....	2....	....	1 & 2	1..2	2....	1....	
Ox. Lead	2..3	2	2	2	3...	2	2....	1.2.3	2	2....	1....	1....	2....	2....	2....	2....	1..2	1....	1....	1
Ox. Copper	1....	2	2	2	3...	2	2....	1....	2	2....	1....	1....	2....	1....	1....	1....	1....	1..2	1..2	1
Ox. Silver	1....	2	2	2	....	2	2....	3....	3	2....	1....	1....	2....	2....	2....	1....	1....	1....	1....	1
Protox. Tin	1....	2	2	...	....	...	2....	1....	2	2....	....	.. .	2....	1..2	....	1....	1....	1..2	1....	2
Perox. Tin	1....	2	...	...	....	...	....	1....	...	2....	1....	....	....	....	....	....	2....	....	1....	
Ox. Bismuth	1....	2	2	2	3...	...	2....	1..2	2	2....	1 & 2	....	2....	2....	....	....	1....	1....	1....	1

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[1] To ascertain the solubility of any salt, find the name of the base in the upright column, and that of the acid in the line at the top; the number placed at the point where the two rows meet, shows whether the salt formed by their combination is soluble or otherwise. The figure 1 means that it is soluble in water; 2, that it is insoluble in water, but soluble in acids; and 3, that it is insoluble in water and acids.

TABLE X.

Showing the Action of Reagents on Oxides and Acids. (Alphabetically arranged.)

1. Metallic Bases. (In Combination.)

Name of Base.	*Symbol.*	*Hydro-sulphuric Acid (Sulphuretted Hydrogen)* (HS), *in an acidified solution.*	*Hydro-sulphate of Ammonia* (NH_4S, HS).	*Carbonate of Soda* (NaO, CO_2).	*Carbonate of Ammonia* ($2NH_4O$, $3CO_2$).	*Potash* (KO).	*Ammonia* (NH_3).	*Ferro-cyanide of Potassium* (K_2Fe Cy_3).	*Blowpipe.*	*Remarks.*
Alumina (see also p. 106).	Al_2O_3.	0	White; insol. in excess.	White; insol. in excess.	White; insoluble.	White; sol. in excess.	White; insol.	0	Blue with nitrate of cobalt.	These precipitates are insoluble in chloride of ammonium.
Antimony, oxide of (see also p. 129).	SbO_3.	Orange red.	Orange red; sol. in excess.	White; sparingly soluble.	White; sp. sol.	White; sp. sol. in excess.	White; insol.	White.	With soda in deoxidizing flame, reduced, and gives off white fumes of oxide.	The chloride decomposed by water.
Baryta (see also p. 101).	BaO.	0	0	White; insol. in excess.	White; insol.	0	0	0	0	Thrown down immediately by sulphates and sulphate of lime.
Bismuth, oxide of (see also p. 138).	BiO_3.	Brown-black.	Brown-black; insol. in excess.	White; insol. in excess.	White; insol.	White; insol.	White; insol.	White.	With soda on charcoal, reduced; brittle bead of metal.	Chloride decomposed by water.

Cadmium, oxide of.	CdO.	Bright yellow.	Bright yellow; insol. in excess.	White; insol. in excess.	White; insol.	White; insol.	White; sol. in excess.	White.	With soda on charcoal reduced and metal volatilized; leaves a reddish-brown deposit.	The yellow sulphide, insol. in hydrosulp. of ammonia is highly characteristic.
Calcium (**Lime**) (see also p. 102).	CaO.	O	O	White; insol. in excess.	White; insol.	O	O	O	Radiates a brilliant light. Gives a red color to the flame.	Oxalate of ammonia causes a white precipitate even in very dilute solutions.
Chromium, oxide of (see also p. 107).	Cr_2O_3.	O	Green.	Green.	Green.	Green; sol. in excess.	Green; insol.	O	Emerald green with fluxes.	The oxide when fused with nitre, gives chromate of potash.
Cobalt, oxide of (see also p. 115).	CoO.	O	Black.	Pink; insol. Bluish on boiling.	Pink; sp. sol. in ex., solution purple.	Blue, becoming greenish; dirty red on boiling.	Blue; sol. in ex. forming a brownish-red sol.	Pale green or gray.	Blue glass with borax in both flames.	Readily distinguished with the blowpipe.
Copper, oxide of (see also p. 135).	CuO.	Black.	Black.	Greenish blue, becoming dark brown on boiling.	Greenish blue, sol. in excess, forming deep blue solution.	Pale blue, becoming dark brown when boiled.	Pale blue, sol. in ex. forming rich blue solution.	Mahogany colored; insol.	With soda on charcoal, reduced. With borax and mic. salt in outer flame, green; in inner flame red.	Precipitated in the metallic state by clean iron.
Glucina.	GlO.	O	O	White; sp. sol.	White; sol. in ex.	White; sol. in ex.	White; insol.	O	With nitrate of cobalt, dark gray or black.	Glucina dissolves in cold solution of carb. ammonia, and is thrown down on boiling.

Name of Base.	*Symbol.*	*Hydrosulphuric Acid* (HS), *in an acidified solution.*	*Hydrosulphate of Ammonia* (NH_4S, HS).	*Carbonate of Soda* (NaO, CO_2).	*Carbonate of Ammonia* ($2NH_4O$, $3CO_2$).	*Potash* (KO).	*Ammonia* (NH_3).	*Ferrocyanide of Potassium* ($K_2, Fe\,Cy_3$).	*Blowpipe.*	*Remarks.*
Gold, teroxide of.	AuO_3.	Black.	Brown black; sol. in excess.	O	Yellow; insol.	Yellowish brown.	Yellow; insol.	O	Reduced.	Thrown down in the form of a brown metallic powder by protosulph. of iron.
Iridium, sesqui-oxide of.	Ir_2O_3.	Slight brown.	Brown; sol.	Brown red; sp. sol.	Bleaches the solution.	Slight brown. The solution becomes first colorless, and subsequently bluish.	As with potash. On exposure to the air, a slight blue precipitate falls.	Solution slowly dis-colored.	Reduced.	The solutions have a deep brown color.
Iron, protoxide of (see also p. 111).	FeO.	O	Black.	White; then green, and ultimately rust-colored.	As with carbonate of soda.	White; becoming green, and on standing, rust-colored.	As with potash; but becoming brown more rapidly.	White, instantly changing to light blue.	With the fluxes in the outer flame, brownish-yellow; in the inner flame, light green.	Deep blue precipitate with ferridcyanide of potassium.
Iron, peroxide of (see also p. 112).	Fe_2O_3.	Yellowish-white precipitate of sulphur.	Black.	Rust-colored.	Rust-colored.	Rust-colored.	Rust-colored.	Deep blue.	As the protoxide.	Black with infusion of gallnuts.
Lead, oxide of (see also p. 133).	PbO.	Black.	Black.	White; insol.	White; insol.	White; sol.	White; insol. None at first with the acetate.	White.	With soda on charcoal, reduced; yellow deposit also formed on the charcoal.	Precipt'd by sol. sulphates, & the precip. blacken'd by hydrosulphate of ammonia. Bright yellow with chromate of potash and iodide of potassium.

LITHIA.	LiO.	O	O	Faint white in concentrated sol.	As carb. soda.	O	O	O	Gives red color to the flame.	Phos. of soda and evapn. gives a white precipitate
MAGNESIA (see also p. 103).	MgO.	O	O	White; insol. on boiling.	O	White; insol.	White; insol.	O	Light pink with nitrate of cobalt.	Crystalline precipit. with phosphate of soda and ammonia. The carbonate and hydrate sol. in chloride of ammonia.
MANGANESE, protoxide of (see also p. 110).	MnO.	O	Flesh-colored.	White; insol.	White; insol.	White; becoming brown.	White; becoming brown.	White;	With soda, a green bead. With borax in outer flame an amethyst bead, which loses its color in the reducing flame.	The presence of ammoniacal salts prevents more or less completely the precipitation of manganese by the alkalies.
MERCURY, suboxide of (see also p. 131).	Hg_2O.	Black.	Black.	Dark gray.	Dark gray.	Black; insol.	Black; insol.	White.	Mixed with soda, and heated in a tube, the metal sublimes.	White precipitate with chlorides, blackened by ammonia. Volatd. or decompd. by heat.
MERCURY, peroxide of (see also p. 132).	HgO.	White, turning to black.	White, turning to black.	Reddish-brown; insol.	White; insol.	Yellow; insol.	White;	White.	As the protoxide.	Volatiliz'd or decompd. by heat. Beautiful scarlet with iodide of potassium.
MOLYBDENUM, oxides of.	MoO and MoO_2.	Brown-black, slowly formed.	Yellowish-brown; sol.	Brown; sol.	Brown; sol.	Brown-black; insol.	Brown-black; insol.	Brown with the binoxide.	With microcosmic salt in outer flame, a green glass.	Most readily distinguished by the blowpipe.

Name of Base.	*Symbol.*	*Hydrosulphuric Acid* (HS), *in an acidified solution.*	*Hydrosulphate of Ammonia* (NH_4S, HS).	*Carbonate of Soda* (NaO,CO_2).	*Carbonate of Ammonia* ($NH_4O, 3CO_2$).	*Potash* (KO).	*Ammonia* (NH_3).	*Ferrocyanide of Potassium* ($K_2,Fe\ Cy_3$).	*Blowpipe.*	*Remarks.*
NICKEL, oxide of (see also p. 114).	NiO.	O	Black.	Pale green; insol.	Pale green; soluble; forming a blue solution.	Pale green; insol.	Pale green; soluble, forming a blue solution.	Pale green.	With soda on charcoal, reduced to a magnetic powder. With borax and mic. salt in outer flame, red glass; becoming colorless on cooling.	Potash throws down a pale green precipitate from the ammoniacal solution.
OSMIUM, deutoxide of.	OsO_2.	Yellowish-brown slowly formed.	Yellowish-brown; insol.	Black, slowly formed. Bluish solution.	Brown after some time.	Black on boiling.	Brown after some time.	O	Osmium is characterized by forming, when heated in the air, osmic acid, which is volatile, and has a very disagreeable smell, causing much inconvenience to the eyes and nose.	
PALLADIUM, protoxide of.	PdO.	Black.	Black; insol.	Brown; sol. Reprecipitated on boiling.	Solution decolorized, but no precipitate.	Yellowish-brown; sol.	Yellowish-brown; sol.	O	Reduced.	Yellowish-white with solution of cyanide of mercury.
PLATINUM, oxide of.	PtO_2.	Brownish-black; formed slowly.	Brownish-black; sol. in large excess.	Yellow with carbonate of potash.	Yellow.	Yellow.	Yellow.	O	Reduced.	Yellow with chloride of ammonium, which is converted by heat into spongy platinum.
POTASH, (see also p. 98).	KO.	O	O	O	O	O	O	O	Violet flame.	White crystalline precipt. with tartaric acid. Yellow with bichloride of platinum.

Rhodium, sesquioxide of.	R_2O_3.	Brown, formed slowly.	Brown; insol.	Yellowish after a time.	Yellowish after a time.	Yellowish-brown on boiling.	Yellowish after a time.	Dark orange.	Reduced.	Many of the compounds have a rose-color.
Silver, oxide of (see also p. 136).	AgO.	Black.	Black.	White; insol.	White; sol.	Pale brown; insol.	Pale brown; sol.	White.	Reduced.	White curdy precipitate with hydrochloric acid and chlorides, which is sol. in ammonia, and insol. in nitric acid.
Soda (see also p. 99).	NaO.	O	O	O	O	O	O	O	Yellow flame.	The only salt which precipitates soda, is the antimoniate of potash. Evaporated with bichloride of platinum, gives yellow needles.
Strontia (see also p. 102).	SrO.	O	O	White; insol.	White; insol.	O	O	O	Carmine flame.	White precipitate with sulphates. Burnt with alcohol, gives carmine flame.
Tin, protoxide of (see also p. 137).	SnO.	Brown-black.	Brown-black.	White; insol.	White; insol.	White; sol.	White; insol.	White.	With soda in reducing flame a malleable bead of metallic tin.	Zinc throws down the metal as a gray sponge.
Tin, peroxide of (see also p. 137).	SnO_2.	Yellow.	Yellow; sol.	White; insol.	White; insol.	White; sol.	White; sp. sol.	White.	Reduced with soda.	The behavior with hydrosulphate of ammonia, and the blowpipe, are characteristic.

Name of Base.	*Symbol.*	*Hydrosulphuric Acid* (HS) *in an acidified solution.*	*Hydrosulphate of Ammonia* (NH_4S, HS).	*Carbonate of Soda* (NaO,CO_2).	*Carbonate of Ammonia* ($2NH_4O$, $3CO_2$).	*Potash* (KO).	*Ammonia* (NH_3).	*Ferrocyanide of Potassium* ($K_2,Fe\ Cy_3$).	*Blowpipe.*	*Remarks.*
URANIUM, sesquioxide of.	U_2O_3.	O (sulphur).	Black.	Yellow; sol.	Yellow; sol.	Yellow; insol.	Yellow; insol.	Reddish-brown.	Yellow glass with borax.	When the precipitate with ammonia is heated, it is converted into the green protoxide.
VANADIUM, binoxide of.	VO_2.	O	Brown-black; sol. in excess, forming a purple solution.	Dirty white.	Gray, passing to brown.	Grayish-white.	Brown.	Yellow.	With borax, yellow in outer flame; in the inner, brown, becoming green when cold.	Many of the solutions have a blue color.
YTTRIA.	YO.	O	White.	White; sp. sol.	White; sp. sol.	White; insol.	White; insol.	White.	Nothing characteristic.	Copious white with oxalic acid.
ZINC, oxide of (see also p. 109).	ZnO.	O.	White.	White; insol.	White; sol.	White; sol.	White; sol.	White.	With soda on charcoal gives a white sublimate of oxide, which is yellow when hot. With nit. cobalt, green.	Behavior with hydrosulphate of ammonia characteristic.
ZIRCONIA.	Zr_2O_3.	O	White.	White after a time.	White; sol.	White; insol.	White; insol.	White.	Bright flame.	Oxalic acid gives a white precipitate.

2. Metallic Oxides having Acid Properties.

Acids (in combination).	*Symbol.*	*Hydrosulphuric acid,* (HS) *in acidified solutions.*	*Hydrosulphate of Ammonia* (NH_4S,HS).	*Chloride of Barium* ($Ba\,Cl$), *(in alkaline salts of the acids).*	*Nitrate of Silver* (AgO, NO_5), *(in alkaline salts of the acids).*	*Nitrate of Lime* ($CaO. NO_5$), *(in alkaline salts of the acids).*	*Hydrochloric acid* (HCl).	*Remarks.*
ANTIMONIOUS ACID.	SbO_4.	Orange.	Orange; sol.	White; sp. sol. in water.	White.	White; sp. sol. in water.	White.	Antimonious acid becomes pale-yellow when heated, and white on cooling. It is insoluble in nitric acid, and difficultly soluble in hot hydrochloric acid.
ANTIMONIC ACID.	SbO_5.	Orange.	Orange; sol.	White.	White.	White.	White.	Insoluble in water and nitric acid. Soluble in hydrochloric acid, from which it is precipitated on the addition of water. When strongly heated, gives off oxygen, and becomes antimonious acid.
ARSENIOUS ACID (see also p. 118).	AsO_3.	Yellow; soluble in alkalies and alkaline sulphides.	Yellow; sol.	White.	Pale-yellow.	White.	O	Volatilizes at a low heat, and condenses in octohedral crystals. The best tests are Marsh's and Reinsch's. (See p. 100.)
ARSENIC ACID (see also p. 128).	AsO_5.	Yellow; soluble in alkalies and alkaline sulphides.	Yellow; sol.	White.	Reddish-brown.	White.	O	Heated with black flux, gives metallic arsenic.
CHROMIC ACID.	CrO_3.	Reduced to oxide, with precipitation of sulphur.	Green.	Yellow.	Reddish-brown.	Yellow in concentrated solutions.	Reduced to oxide, with evolution of chlorine.	Is decomposed by heat and by deoxidizing agents, into oxide of chromium. Salts of lead throw down a yellow precipitate.

Acids (*in combination*).	*Symbol.*	*Hydrosulphuric acid*, (HS) *in acidified solutions.*	*Hydrosulphate of Ammonia* (NH_4S,HS).	*Chloride of Barium* ($Ba Cl$), (*in alkaline salts of the acids*).	*Nitrate of Silver* (AgO, NO_5), (*in alkaline salts of the acids*).	*Nitrate of Lime* (CaO, NO_5), (*in alkaline salts of the acids*).	*Hydrochloric acid* (HCl).	*Remarks.*
MANGANIC ACID.	MnO_3.	The same.	Flesh-colored.	○	Black (oxide).	Black.	Solution becomes red, and chlorine is evolved.	Converted by acids into hypermanganic acid and peroxide of manganese; the color of the solution changing from green to red.
MOLYBDIC ACID.	MoO_3.	Brown.	Brown; sol.	White.	White.	White.	White.	With microcosmic salt before the blowpipe, gives a dark blue glass, which becomes green on cooling. When strongly heated, molybdic acid volatilizes and condenses in crystals.
TUNGSTIC ACID.	WO_3.	Slight turbidity.	Brown; sol.	White.	White.	White.	White; insoluble.	Does not volatilize when heated. Has a pale yellow color, and is insoluble in water and acids. Blue with hydrochloric acid and zinc.
VANADIC ACID.	VO_3.	Gray.	Brown; sol.	Orange.	Yellow.	○	Chlorine evolved.	When treated with hydrochloric acid, the mixture is capable of dissolving gold-leaf. Vanadic acid in solution is readily deoxidized, forming a blue liquid.

3. NON-METALLIC ACIDS.

Acids (*neutralized*).	*Symbol.*	*Nitrate of Baryta* (BaO,NO_5).	*Nitrate of Silver* (AgO,NO_5).	*Nitrate of Lime* (CaO,NO_5).	*Acetate of Lead* ($PbO, C_4H_3O_3$).	*Remarks.*
BORACIC ACID (see also p. 150).	BO_3.	White.	White.	White.	White.	Slightly volatile in the presence of aqueous vapor. Turns turmeric-paper brown, and blue litmus port-wine color. Gives green color to the flame of alcohol.
BROMIC ACID.	BrO_5.	White.	White.	○	White.	The bromates are decomposed by heat into bromides and oxygen. Sulphuric acid disengages bromine.

CARBONIC ACID (see also p. 152).	CO_2.	White.	White.	White.	White.	The carbonates are readily decomposed by acids, carbonic acid gas being given off with effervescence, which, when passed into lime-water, gives a white precipitate.
CHLORIC ACID (see also p. 147).	ClO_5.	O	O	O	O	All the chlorates are soluble in water. At a red heat they are converted into chlorides, oxygen being given off.
HYDRIODIC ACID (see also p. 148).	HI.	O	Pale yellow.	O	Bright yellow.	The iodides evolve iodine when heated with nitric or sulphuric acid. With chlorine water and starch, they give a dark purple precipitate.
HYDROBROMIC ACID.	HBr.	O	Yellowish.	O	White.	The bromides, when heated with nitric acid, evolve bromine.
HYDROCHLORIC ACID (see also p. 146).	HCl.	O	White.	O	White.	The chlorides, when heated with sulphuric acid and peroxide of manganese, evolve chlorine.
HYDROCYANIC ACID.	H,C_2N.	O	White.	O	White.	With a mixture of a protosalt, and persalt of iron, the alkaline cyanides give a precipitate of Prussian blue.
HYDROFLUORIC ACID.	HF.	White.	O	White.	O	The fluorides, when moistened with sulphuric acid, give off fumes which corrode glass.
HYDROSELENIC ACID.	HSe.	O	Black.	O	Black.	The selenides, when heated in the outer flame of the blowpipe, evolve the odor of selenium, resembling that of putrid horseradish.
HYDROSULPHURIC ACID (see also p. 142).	HS.	O	Black.	O	Black.	Most of the sulphides, when treated with an acid, evolve hydrosulphuric acid, which smells like rotten eggs.
HYPOSULPHUROUS ACID.	S_2O_2.	White.	White, becoming brown.	O	White.	The hyposulphites are decomposed by hydrochloric acid; sulphur is precipitated, and sulphurous acid set free.
HYPOSULPHURIC ACID.	S_2O_5.	O	O	O	O	The hyposulphates are decomposed without deposition of sulphur, when boiled with hydrochloric acid; sulphurous and sulphuric acids are formed.

Acids (neutralized).	*Symbol.*	*Nitrate of Baryta* (BaO,NO_5).	*Nitrate of Silver* (AgO,NO_5).	*Nitrate of Lime* (CaO,NO_5).	*Acetate of Lead* ($PbO, C_4H_3O_3$).	*Remarks.*
IODIC ACID.	IO_5.	White.	White.	White.	White.	The iodates are decomposed by heat into iodides and oxygen.
NITRIC ACID (see also p. 141).	NO_5.	O	O	O	O	When mixed with sulphuric and hydrochloric acids, the nitrates dissolve gold-leaf. With copper filings and sulphuric acid, orange fumes are given off.
PERCHLORIC ACID.	ClO_7.	O	O	O	O	The perchlorates are resolved by heat into chlorides and oxygen. They are not decomposed in the cold by hydrochloric or sulphuric acid; thus differing from the chlorates.
PHOSPHORIC ACID (Tribasic) (see also p. 144.)	PO_5.	White.	Yellow.	White.	White.	The soluble phosphates give with salts of magnesia, when ammonia is present, a white crystalline precipitate.
PHOSPHOROUS ACID.	PO_3.	White.	White, becoming brown.	White.	White.	The hydrated phosphites are decomposed when heated in a tube; hydrogen or phosphuretted hydrogen is given off, and phosphates are formed.
SELENIC ACID.	SeO_3.	White.	White.	White.	White.	The seleniates are decomposed by boiling with hydrochloric acid; chlorine is evolved, together with selenious acid.
SELENIOUS ACID.	SeO_2.	White.	White.	White.	White.	Metallic zinc or sulphurous acid causes the precipitation of selenium from acidified solutions of the selenites.
SILICIC ACID. (see also p. 151).	SiO_3.	White.	Pale yellow.	White.	White.	When a soluble silicate is evaporated to dryness with hydrochloric acid, it is decomposed, and the silica remains insoluble.
SULPHURIC ACID (see also p. 142).	SO_3.	White.	White, crystalline.	White, crystalline.	White.	Most of the sulphates, when heated with charcoal, are converted into sulphides, which, when moistened with hydrochloric acid, evolve hydrosulphuric acid.
SULPHUROUS ACID.	SO_2.	White.	White.	White.	White.	The sulphites are decomposed by sulphuric acid, sulphurous acid being given off, without the deposition of sulphur.

4. ORGANIC ACIDS.

Acids (neutralized).	Symbol.	Chloride of Calcium ($CaCl$).	Perchloride of Iron (Fe_2Cl_3).	Nitrate of Baryta (BaO,NO_5).	Nitrate of Silver (AgO,NO_5).	Acetate of Lead ($PbO,C_4H_3O_3$).	Remarks.
ACETIC ACID (see also p. 154).	$HO, C_4H_3O_3$.	O	Red color.	O	White; crystalline in concentrated solutions.	O	The acetates, when warmed with sulphuric acid, give off the smell of vinegar. Acetic acid boiled with an excess of protoxide of lead, forms the subacetate, which is alkaline to test-paper.
BENZOIC ACID (see also p. 158).	$HO, C_{14}H_5O_3$.	O	Brownish-yellow.	O	Crystalline in concentrated neutral solutions.	White in concentrated neutral solutions.	Solutions of the benzoates, when treated with sulphuric acid, give a crystalline precipitate of benzoic acid.
CITRIC ACID (see also p. 157).	$3HO, C_{12}H_5O_{11}$.	White.	O	White.	White.	White.	With protonitrate of mercury, a white precipitate, which becomes gray.
FORMIC ACID.	HO, C_2HO_3.	O	Red color.	O	White, becoming black, especially when warmed.	O	The formiates, when warmed with sulphuric acid, do not blacken, and give off carbonic oxide gas.
MALIC ACID.	$2HO, C_8H_4O_8$.	White on the addition of alcohol.	O	White.	White; becoming gray.	White precipitate, that melts in boiling water.	Malate of lead dissolves in hot dilute acetic acid, and crystallizes on cooling in fine needles. Malic acid is decomposed by heat, into malæic and fumaric acids.
OXALIC ACID (see also p. 153).	HO, C_2O_3.	White.	O	White crystalline.	White.	White.	Neither the acid nor the oxalates are blackened by strong sulphuric acid, but give off carbonic acid and carbonic oxide gases.
SUCCINIC ACID.	$2HO, C_8H_4O_6$.	O	Reddish-brown.	O	White on standing.	White.	A mixture of chloride of barium, ammonia, and alcohol, gives a white precipitate of succinate of baryta.
TARTARIC ACID (see also p. 156).	$2HO, C_8H_4O_{10}$.	White.	O	White.	White.	White.	Added in excess to potash, gives a crystalline precipitate of the bitartrate.

LIST OF SALTS, &c.

WHICH MAY BE EXAMINED FOR PRACTICE.

I. Exercises in the use of the Blowpipe (p. 71).

(a.) One constituent to be detected.

1. Sulphur.
2. Arsenious acid.
3. Calomel.
4. Chloride of ammonium.
5. Oxide of zinc.
6. Binoxide of tin.
7. Oxide of copper.
8. Oxide of lead.
9. Peroxide of iron.
10. Oxide of nickel.
11. Oxide of cobalt.
12. Oxide of chromium.
13. Binoxide of manganese.
14. Carbonate of soda.
15. Bicarbonate of potash.
16. Carbonate of lime.
17. Carbonate of baryta.
18. Silica.
19. Boracic acid.
20. Charcoal.

(b.) Two constituents to be detected.

21. Sulphate of iron.
22. Trisnitrate of bismuth.
23. Sulphide of arsenic.
24. Vermilion.
25. Iodide of lead.
26. Chlorate of potash.
27. Chloride of sodium.
28. Fluoride of calcium.
29. Sulphide of copper.
30. Sulphate of zinc.
31. Pipe clay.
32. Sulphate of magnesia.
33. Argentiferous lead.[1]
34. Sulphide of antimony.
35. Iron pyrites.
36. Chromate of potash.
37. Nitrate of strontia.
38. Sulphate of baryta.
39. Borax.
40. Alloy of lead and antimony.[2]
41. Arsenite of copper.
42. Acetate of lead.

(c.) Three constituents to be detected.

43. Potash-alum.
44. Ammonia-alum.
45. Tartar-emetic.
46. Arsenical pyrites.

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[1] A pound of lead should be fused with a little borax, and 200 grs. of silver thoroughly dissolved in it. The alloy is then granulated by pouring into water, or better, cast into a thin plate, on a stone, and cut up into fragments.

[2] An alloy of 4 lead and 1 antimony may be used.

II. Exercises in Qualitative Analysis.

(*a.*) *Simple Salts, &c., soluble in Water* (*p.* 163).

1. Chloride of barium.
2. Sulphate of soda.
3. Chloride of ammonium.
4. Sulphate of magnesia.
5. Chloride of calcium.
6. Nitrate of strontia.
7. Sulphate of chromium.
8. Sulphate of zinc.
9. Sulphate of manganese.
10. Protosulphate of iron.
11. Perchloride of iron.
12. Sulphate of nickel.
13. Nitrate of cobalt.
14. Protonitrate of mercury.
15. Perchloride of mercury.
16. Acetate of lead.
17. Sulphate of copper.
18. Nitrate of silver.
19. Phosphate of soda.
20. Iodide of potassium.
21. Biborate of soda.
22. Nitrate of potash.
23. Chlorate of potash.
24. Carbonate of soda.
25. Arsenious acid.

(*b.*) *Simple Salts, &c., insoluble in Water, but soluble in Acids* (*p.* 163).

26. Carbonate of magnesia.
27. Phosphate of lime.
28. Carbonate of baryta.
29. Metallic zinc.
30. Carbonate of strontia.
31. Sulphide of iron.
32. Protoxide of lead.
33. Sulphide of antimony.
34. Black oxide of copper.
35. Carbonate of lime.
36. Oxide of bismuth.
37. Metallic tin.

(*c.*) *Simple Salts, &c., insoluble in Water and Acids* (*p.* 166).

38. Sulphate of baryta.
39. Sulphate of strontia.
40. Chloride of lead.
41. Sulphate of lead.
42. Silica.
43. Chloride of silver.
44. Fluoride of calcium.
45. Silicate of alumina (pipe clay).

(*d.*) *Mixed Salts, &c., soluble in Water* (*p.* 192).[1]

46. Sulphate of copper.
    Sulphate of iron.
47. Nitrate of lead.
    Nitrate of silver.
48. Sulphate of manganese.
    Sulphate of magnesia.
49. Alum.
50. Phosphate of soda and ammonia.
51. Tartar-emetic.
52. Carbonate of soda.
    Sulphite of soda.
53. Commercial cyanide of potassium (containing carbonate of potash).
54. Biborate of soda.
    Chloride of ammonium.
55. Chlorate of potash.
    Chloride of sodium.
56. Iodide of potassium.
    Carbonate of potash.
57. Sulphate of copper.
    Sulphate of iron.
    Sulphate of nickel.
58. Sulphate of zinc.
    Sulphate of copper.
59. Chrome alum.
60. Chloride of barium.
    Nitrate of strontia.

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[1] The constituent placed first should always form the greater part of the mixture.

(*e.*) *Mixed Salts, &c., not entirely soluble in Water, but soluble in Acids* (*p.* 192).

61. Carbonate of lime.
Carbonate of magnesia.
62. Binoxide of manganese.
Peroxide of iron.
Carbonate of baryta.
63. Sulphide of iron.
Arsenious acid.
64. Peroxide of iron.
Oxide of cobalt.
Oxide of nickel.
65. Calomel.
Corrosive sublimate.
66. Acetate of lead.
Verdigris.
67. Sulphide of zinc.
Sulphide of lead.
Sulphide of iron.
68. Peroxide of iron.
Binoxide of manganese.
Phosphate of lime.
Arsenious acid.
69. Sulphide of antimony.
Sulphide of arsenic.
Sulphide of lead.
70. Oxalate of ammonia.
Sulphate of lime.
71. Chloride of lime (bleaching powder).
72. Brass.
73. German silver.
74. Phosphate of lime.
Alum.

(*f.*) *Mixed Salts, &c., not entirely soluble in Water or Acids* (*p.* 192).

75. Sulphate of baryta.
Sulphate of lime.
76. Pipe clay.
Chrome iron ore.
77. Gunpowder.
78. Sulphide of lead.
Binoxide of tin.
Silica.
Fluor spar.
79. Sulphate of lead.
Chloride of silver.
80. Vermilion.
Red lead.
81. Prussian blue.
Carbonate of baryta.
82. Bronze.
83. Pewter.
84. Type metal.
85. Fusible metal.
86. Crown glass.
87. Flint glass.
88. Red earthenware.

GLOSSARY OF CHEMICAL TERMS.[1]

Absorption, from *absorbeo*, to suck up; the act of imbibing a liquid.

Acetic acid, from *acetum*, vinegar; the acid contained in vinegar.

Aeriform, from ἀὴρ, the air, and *forma*, a form; having the form or properties of air.

Affinity, from *ad*, to, and *finis*, a boundary; relationship; the force which causes particles of dissimilar kinds of matter to combine together, so as to form new matter.

Albumen, -inous, from *albumen*, the white of an egg; an important animal principle. The white of an egg consists chiefly of albumen and water, contained in a cellular tissue.

Alcohol, from an Arabic word; the intoxicating principle of spirituous liquors.

Alkali, a soluble body, with a hot caustic taste, which possesses the power of destroying or neutralizing acidity. The term is derived from the Arabic article, *al*, and *kali*, the Arabic name of a plant, from the ashes of which one of the most important alkalies (potash) is obtained.

Amalgam, from ἅμα, together, and γαμέω, to marry; a term signifying the union of any metal with mercury, which has the property of dissolving several of the metals.

Amorphous, from ἀ, not, and μορφὴ, a form; not possessing any regular form.

Analogue, that which is the counterpart of another.

Analogy, -ical, and -ous, from ἀνὰ, among, and λόγος, a relation or proportion; a likeness or resemblance between things, with regard to their circumstances or effects.

Analysis, from ἀνὰ, among, and λύω, to loosen; the separation of a substance into its component parts.

Angle, from *angulus*, a corner; the inclination of two straight lines to each other, which meet together, but are not in the same straight line.

Anhydrous, from ἀ, not, and ὕδωρ, water; containing no water.

Antiseptic, from ἀντὶ, against, and σήπω, to putrefy; possessing the power of preventing or retarding putrefaction.

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[1] Many of the definitions are taken almost verbatim from Daniell's Chemical Philosophy.

Aqua Regia, *i. e.*, **Regal Water**, a mixture of nitric and hydrochloric acids; so called from its property of dissolving gold, which was held by the alchemists to be the king of the metals.

Aqueo, from *aqua*, water; when prefixed to a word, denotes that water enters into the composition of the substance which it signifies, as *aqueo-sulphuric acid* (HO,SO_3). [*Obsolete.*]

Athermanous, from ἀ, not, and θέρμες, heat; that through which heat will not pass, is said to be *athermanous*.

Atmosphere, from ἀτμὸς, vapor, and σφαῖρα, a sphere; commonly used to denote the sphere of air which surrounds the globe.

Atom, **-ic**, from ἀ, not, and τέμνω, to cut; a minute particle of matter, not susceptible of further division.

Attraction, **-ive**, from *ad*, to, and *traho*, to draw; the tendency which bodies have to approach or unite with each other.

Azote, from ἀ, not, and ζωὴ, life; another name for nitrogen; so called because it is incapable of supporting respiration.

Barium, from βαρὺς, heavy; the metallic base of baryta.

Barometer, from βάρος, weight, and μέτρον, a measure; an instrument for measuring the varying pressure of the atmosphere.

Baryta, a compound of oxygen and the metal *barium* (BaO), possessing alkaline properties.

Bibulous, from *bibo*, to drink; that which has the property of drinking in, or absorbing, moisture.

Boron, a dark, olive-colored elementary substance, obtained from boracic acid, insoluble in water, and a non-conductor of electricity.

Bromine, from βρῶμος, a strong odor; an elementary liquid of a reddish-brown color and suffocating smell; in chemical properties it strongly resembles chlorine.

Caloric, from *calor*, heat; an imaginary fluid substance, supposed to be diffused through all kinds of matter, and the sensible effect of which is called *heat*.

Capillary, from *capillus*, a hair; resembling, or having the tubular form of hairs.

Capsule, from *capsula*, a little chest; a small shallow cup.

Carbon, from *carbo*, a coal; the chemical name for charcoal.

Caustic, from καίω, to burn; possessing the power of burning.

Chemistry, **-ical**, from an Arabic word, signifying the knowledge of the substance or constitution of bodies; the science whose object it is to examine the constitution of bodies.

Chlorine, from χλωρὸς, green; a greenish colored gas, of a pungent suffocating smell, and possessing chemical properties nearly allied to those of oxygen.

Cleavage, **Plane of**, the plane in which crystals have a tendency to separate.

Cohesion, from *con*, together, and *hæreo*, to stick; the power which causes the particles of a body to cling together and resist separation.

Combustion, from *comburo*, to burn; the disengagement of light and heat, which frequently accompanies chemical combination.

Conduction, from *con*, together, and *duco*, to lead; the power of transmitting heat or electricity, without change in the relative position of the particles of the conducting body.

Congelation, from *con*, together, and *gelo*, to freeze; the process of freezing.

Constituent, from *constituo*, to put together; that of which anything consists, or is made up.

Contraction, from *con*, together, and *traho*, to draw; the state of being drawn into a narrow compass, or becoming smaller.

Convex, from *con*, together, and *veho*, to carry; curved outwardly or protuberant.

Corpuscular, from *corpus*, a body; composed of, or relating to, atoms.

Crystallography, from κρύσταλλος, a crystal or ice, and γράφω, to describe; the science which treats of crystals.

Crystallization; the formation of crystals during the passage of certain substances from a fluid to a solid state.

Cube, -ic; a solid figure contained by six equal squares.

Cyanogen, from κύανος, blue, and γεννάω, to produce; a colorless gas composed of carbon and nitrogen (C_2N). Its chemical properties much resemble those of chlorine; it derives its name from the circumstance of its entering into the composition of prussian-blue.

Cyanide; a compound of cyanogen with a metal is called a cyanide, as cyanide of potassium (K,C_2N).

Decomposition; the resolution of a compound substance into its component parts.

Decrepitation, from *de*, from, and *crepito*, to crackle; the crackling noise which certain salts make when heated, usually caused by the sudden escape of water.

Deflagration, from *deflagro*, to burn; burning very rapidly.

Deliquescence, from *deliqueo*, to melt; a gradual melting or dissolving, caused by the absorption of water from the atmosphere.

Density, from *densus*, thick; vicinity or closeness of particles; specific weight.

Deoxidize, to deprive of oxygen.

Detonation, from *detono*, to thunder; explosion accompanied with noise.

Diaphonous, from διὰ, through, and φαίνω, to shine; that which allows a passage to the rays of light, but disperses them so as to prevent direct vision.

Diathermanous, from διὰ, through, and θερμὸς, heat; that through which heat will pass, is said to be *diathermanous*.

Dimorphous, from δὶς, twice, and μορφὴ, a form; having two distinct crystalline forms.

Disintegration, from *dis*, meaning separation, and *integer*, whole; a complete separation of particles.

Distillation, a separation drop by drop; the process by which a fluid is separated from another substance by being first converted into vapor, and afterwards condensed drop by drop.

Dodecahedron, from δώδεκα, twelve, and ἕδρα, a base or side; a solid figure contained by twelve equal sides.

Ebullition, from *ebullio*, to boil; the act of boiling.

Effervescence, the escape of bubbles of gas formed in a liquid, as when marble is decomposed by hydrochloric acid.

Efflorescence, from *effloresco*, to blow as a flower; the formation of small crystals on the surfaces of bodies, in consequence of the abstraction of water from them by the atmosphere.

Electricity, from ἤλεκτρον, amber; the name of a power of matter, which produces a variety of peculiar phenomena, the first of which were observed in the mineral substance called amber; the laws, hypotheses, and experiments by which they are explained and illustrated constitute the science of electricity.

Electrode, from ἤλεκτρον, electricity, and ὁδὸς, a way; the point at which an electric current enters or quits the body through which it passes.

Electrolysis, -lyte, from ἤλεκτρον, electricity, and λύω, to loosen; the act of decomposing bodies by electricity.

Element, from *elementum*, an element; that which cannot be resolved into two or more parts, and contains but one kind of ponderable matter.

Empyreumatic, from ἐν, in, and πῦρ, fire; having the taste or smell of burnt animal or vegetable substances.

Endosmose, from ἔνδον, within, ὠσμὸς, the act of pushing; a flowing from the outside to the inside.

Equivalent, from *æquus*, equal, and *valeo*, to be worth; equal in value, or in the power of combining with other substances.

Evaporation, from *e*, out, and *vapor*, vapor; the conversion of a liquid into vapor.

Exosmose, from ἔξω, without, and ὠσμὸς, the act of pushing; a flowing from the inside to the outside.

Expansion, from *expando*, to open out; the enlargement or increase in the bulk of bodies, which is produced by heat.

Experiment, from *experior*, to attempt, to try; something done in order to discover an uncertain or unknown effect.

Explosion, from *ex*, out, and *plaudo*, to utter a sound; a sudden expansion of an elastic fluid, with force and a loud report.

Fermentation, from *fermentum*, that which is light and puffy; originally applied to the process by which alcohol is formed in saccharine liquids. A metamorphosis or transposition of particles caused by contact with a substance in a state of chemical change.

Ferruginous, from *ferrum*, iron; belonging to, or containing iron.

Filter, a strainer.

Fluorine, from *fluo*, to flow; an elementary principle contained in fluor spar, which is so called from its acting as a flux in the working of certain minerals.

Flux, from *fluo*, to flow; that which assists in the fusion of other substances when mixed with it.

Focus, from *focus*, a fire place; a point in which a number of rays of light or heat meet, after being refracted or reflected.

Gas, a permanent aëriform fluid.

Gelatinous, from *gelo*, to freeze; resembling jelly.

Gravity, from *gravis*, heavy; the natural tendency of bodies to fall towards a centre, usually the centre of the earth.

Gravity, Specific; the relative gravity or weight of a body, considered with regard to an equal bulk of some other body, which is assumed as a standard of comparison.

Heterogeneous, from ἕτερος, different, and γένος, kind; different in nature and properties.

Homogeneous, from ὁμὸς, like, and γένος, kind; alike in nature and properties.

Hydrate, from ὕδωρ, water; any substance which contains water chemically combined.

Hydrogen, from ὕδωρ, water, and γεννάω, to produce; an inflammable, colorless, and aëriform fluid; the lightest of all known substances, and one of the elements of water.

Hydro; when prefixed to the name of a chemical substance, denotes that hydrogen enters into the composition of the substance which it signifies.

Hydrostatics, from ὕδωρ, water, and στατὸς, poised; the branch of Natural Philosophy which treats of the pressure and equilibrium of non-elastic fluids, and also of the weight, pressure, &c., of solids immersed in them.

Hypo, from ὑπὸ, under; when prefixed to a word, denotes an inferior quantity of some ingredient which enters into the composition of the substance which it signifies.

Hypothesis, from ὑπὸ, under, and τιθημι, to place; a principle supposed, or taken for granted, in order to prove a point in question.

Ignite, from *ignis*, fire; to heat a substance to redness; to set on fire.

Imponderable, from *in*, not, and *pondero*, to weigh; that which has no perceptible weight.

Incandescent, from *incandesco*, to grow white; white or glowing with heat.

Increment, from *incresco*, to increase; the quantity by which anything increases or becomes greater.

Induction, electrical, from *in*, to, and *duco*, to lead; the effect produced by the tendency of an insulated electrified body, to excite an opposite electric state in neighboring bodies.

Inertia, from *inertia*, inactivity; the disposition of matter to remain in its state of rest or motion.

Inflammable, from *in*, and *flamma*, a flame; capable of burning with a flame.

Insulation, from *insula*, an island; when a body containing a quantity of free heat or electricity is surrounded by non-conductors, it is said to be *insulated*.

Interstices, from *interstitium*, a break or interval; the unoccupied spaces between the molecules of bodies.

Iodide; a compound of iodine and a metal.

Iodine, from ἴον, a violet, and εἶδος, the form or likeness; a soft opaque elementary substance, which, when heated, sublimes in the form of a violet-colored vapor.

Isomeric, from ἴσος, equal, and μέρος, a part; substances which consist of the same ingredients, in the same proportions, and yet differ essentially in their properties, are called *isomeric*.

Laminæ, from *lamina*, a thin plate; extremely thin plates, of which some solid bodies are composed.

Levigation, from *lævis*, smooth; the reducing of hard bodies to a very fine powder, by grinding with water.

Lignin, from *lignum*, wood; an organic principle of which the fibres of vegetables are mainly composed.

Litmus, a blue pigment obtained from the lichen *rocçella;* it is a most delicate test for acids, which turn it red.

Malleable, from *malleus*, a hammer; that which is capable of being spread out by hammering.

Metallurgy, from μέταλλον, a metal, and ἔργον, a work; the art of working metals, and separating them from their ores.

Molecules, -ar, a diminutive from *mòles*, a mass; the infinitely small material particles, of which bodies are conceived to be aggregations.

Mucilaginous; resembling mucilage or gum.

Murexide, from *murex*, a fish affording a purple dye; a beautiful purple compound, resulting from the decomposition of uric acid by means of nitric acid.

Nascent, from *nascor*, to be born; in the moment of formation.

Nitrogen, from νίτρον, nitre, and γεννάω, to produce; a colorless elementary gas, devoid of taste and smell; it is one of the constituents of the atmosphere, and also of nitric acid, from which latter circumstance it derives its name.

Nitrogenous; containing nitrogen in combination.

Nucleus, from *nucleus*, a kernel; the central parts of a body, which are supposed to be firmer, and separated from the other parts, as the kernel of a nut is from the shell; also, the point about which matter is collected.

Octohedron, -al, from ὀκτὼ, eight, and ἕδρα, a side; a solid figure contained by eight equal and equilateral triangles.

OLEFIANT GAS, from *oleum*, oil, and *fio*, to become; a colorless gas, composed of carbon and hydrogen (C_4H_4), which derives its name from its property of forming an oil-like liquid with chlorine.

ORGANIC MATTER, from ὄργανον, an organ; matter of which the organic parts or juices of plants and animals are composed, or which is derived from such parts by the action of chemical agents, is called *organic*.

OXIDE; a compound of oxygen with a metal or non-metallic body, not having acid properties.

OXIDIZE; to combine with oxygen.

OXYGEN, from ὀξύς, acid, and γεννάω, to produce; a colorless elementary gas, which was formerly supposed to be the universal acidifying principle.

PELLICLE, a diminutive from *pellis*, a skin or crust; a thin crust formed on the surface of a solution.

PERCOLATE, from *per*, through, and *colo*, to strain; to strain through.

PERMEATE, from *permeo*, to pass through; to penetrate.

PHENOMENON, from φαίνομαι, to appear; an appearance which is more or less remarkable.

PHILOSOPHY; from φιλέω, to love, and σοφία, wisdom; the study or knowledge of nature or morality, founded on reason and experience; the word originally implying "A love of wisdom."

PHLOGISTON, from φλέγω, to burn; a name given by the older chemists to an imaginary substance, which was considered as the principle of inflammability.

PHOSPHORUS, from φῶς, light, and φέρω, to produce; a highly inflammable elementary substance, obtained from calcined bones, which emits light when placed in the dark, owing to its undergoing a slow combustion.

PHYSICS, -ICAL, from φύσις, nature; the science of natural bodies, their phenomena, causes, and effects, with their affections, motions, and operations.

PNEUMATICS, from πνεῦμα, air; that branch of Natural Philosophy which treats of the weight, elasticity, and other properties of aëriform fluids.

POLARITY; the opposition of two equal forces in bodies, similar to that which confers the tendency of magnetized bodies to point towards the magnetic poles.

POLARIZED LIGHT; light which, by reflection or refraction at a certain angle, or by refraction in certain crystals, has acquired the property of exhibiting opposite effects in planes at right angles to each other, is said to be *polarized*.

PORES, from πόρος, a passage; the small interstices between the solid particles of bodies.

PRECIPITATION, from *præcipito*, to fall suddenly; the formation and separation of a solid substance in a liquid.

PRODUCT, from *pro*, forth, and *duco*, to draw; anything formed from the elements of another by an operation.

Pyro, from πῦρ, fire; when prefixed to a word, denotes that the substance which it signifies, has been formed at a high temperature.

Qualitative; regarding the properties of a body, and the kinds of matter of which it is composed, without reference to quantity.

Quantitative; regarding quantities.

Radiation, from *radius*, a ray; the shooting forth in all directions from a centre.

Rarefaction, from *rarus*, rare, and *facio*, to make; the act of causing a substance to become less dense; it also denominates the state of this lessened density.

Rectification; the process of drawing anything off by distillation, in order to obtain it in a state of greater purity.

Refraction, from *re*, back, and *frango*, to break; the deviation of rays of light or heat from their direct course, when passing through media of different densities.

Refrigeration, from *re*, again, and *frigus*, cold; the act of cooling.

Repulsion, from *re*, back, and *pello*, to drive; that property in certain bodies, whereby they mutually tend to recede from each other.

Salifiable bases, from *sal*, salt, and *fio*, to become; bodies capable of combining with acids to form salts.

Sapid, from *sapio*, to taste of; possessing the power of exciting the organs of taste.

Saturation, -ated, from *satur*, full; the solution of one body in another until the receiving body can contain no more. A solution is said to be saturated with an acid or an alkali, when the latter is added in sufficient quantity to render it neutral, and *supersaturated* when the point of neutrality has been exceeded.

Solution, from *solvo*, to loosen or melt; any liquid which contains another substance dissolved in it.

Solvent; any substance which will dissolve another.

Specific, from *species*, a particular sort or kind; that which denominates any property which is not general, but is confined to an individual species.

Specific gravity; see **Gravity, specific.**

Sublimation, from *sublimis*, high; the act of raising into vapor by means of heat, and condensing in the upper part of the vessel in a solid state.

Sulphide (Sulphuret); a combination of a metal with sulphur.

Supersaturate; see Saturation.

Ternary, from *ter*, thrice; containing three units.

Tetrahedron, from τέσσαρες, four, and ἕδρα, a base or side; a solid figure contained by four equal and equilateral triangles.

Transparent; a term to denote the quality of a substance which not only admits the passage of light, but also of the vision of external objects.

Triturate, from *trituro*, to thresh; to reduce to powder.

Vacuum, from *vacuus*, empty; a space empty, and devoid of all matter.

Volume, from *volumen*, a roll; the apparent space occupied by a body.

Weight; the pressure which a body exerts vertically downwards in consequence of the action of gravity.

Zero; the numeral 0, which fills the blank between the ascending and descending numbers of a series.

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